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BULLETIN NO. 62.

U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

PROCEEDINGS

OF THE

SEVENTEENTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

WASHINGTON, D. C.

NOVEMBER 16, 17, AND 19, 1900.

EDITED BY

HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.

1862 1889

WASHINGTON:
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LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Division of Chemistry,
Washington, D. C., January 28, 1901.

Sir: I have the honor to transmit to you herewith the manuscript of the proceedings of the seventeenth annual meeting of the Association of Official Agricultural Chemists, with the request that it be published as Bulletin No. 62 of the Division of Chemistry.

H. W. Wiley,
Chief of the Division of Chemistry and Secretary of the Association of Official Agricultural Chemists.

Hon. James Wilson,
Secretary of Agriculture.

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PROCEEDINGS OF THE SEVENTEENTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

FRIDAY—MORNING SESSION.

The seventeenth annual convention of the Association of Official Agricultural Chemists was held in Washington, D. C., beginning November 16, 1900. The meeting was called to order by the president, Mr. B. W. Kilgore, in the lecture hall of the Columbian University.

MEMBERS AND VISITORS PRESENT.

The following members and visitors were in attendance:

Allen, W. M., Assistant State Chemist, Raleigh, N. C.
Anderson, J. T., Agricultural Experiment Station, Auburn, Ala.
Bache, A. W., U. S. Department of Agriculture, Washington, D. C.
Bartlett, J. M., Agricultural Experiment Station, Orono, Me.
Beal, W. H., U. S. Department of Agriculture, Washington, D. C.
Bergami, Francis, Chemist, Baugh & Sons Co., Philadelphia, Pa.
Bigelow, W. D., U. S. Department of Agriculture, Washington, D. C.
Brackett, R. N., Agricultural Experiment Station, Clemson College, S. C.
Cameron, F. K., U. S. Department of Agriculture, Washington, D. C.
Carpenter, B. F., Chemist, Virginia-Carolina Chemical Company, Richmond, Va.
Carr, Oma, care J. P. Heald & Co., Lynchburg, Va.
Church, C. G., Maryland Agricultural College, College Park, Md.
Covert, J. R., U. S. Department of Agriculture, Washington, D. C.
Davidson, R. J., Agricultural Experiment Station, Blacksburg, Va.
Dyer, B., London, England.
Edelen, George S., Maryland Agricultural College, College Park, Md.
Ewell, E. E., U. S. Department of Agriculture, Washington, D. C.
Fields, John, Agricultural Experiment Station, Stillwater, Okla.
Fraps, G. S., Agricultural Experiment Station, Raleigh, N. C.
Frear, William, Agricultural Experiment Station, State College, Pa.
Gough, T. R., Maryland Agricultural College, College Park, Md.
Hand, W. F., Mississippi Agricultural College, Agricultural College, Miss.
Hardy, J. C., Mississippi Agricultural College, Agricultural College, Miss.
Herff, B. von, German Kali Works, New York, N. Y.
Hills, J. L., Agricultural Experiment Station, Burlington, Vt.
Hite, B. H., Agricultural Experiment Station, Morgantown, W. Va.
Holland, E. B., Hatch Experiment Station, Amherst, Mass.

Huston, H. A., Agricultural Experiment Station, Lafayette, Ind.
Jones, C. H., Agricultural Experiment Station, Burlington, Vt.
Kilgore, B. W., State Chemist, Raleigh, N. C.
Krug, W. H., U. S. Department of Agriculture, Washington, D. C.
Lamb, F. C., Raleigh, N. C.
Langworthy, C. F., U. S. Department of Agriculture, Washington, D. C.
Laughlin, J. L., Maryland Agricultural College, College Park, Md.
Le Clerc, J. A., Agricultural Experiment Station, Geneva, N. Y.
Leech, A. E., State Board of Health, Boston, Mass.
McCandless, J. M., State Chemist, Atlanta, Ga.
McDonnell, H. B., Agricultural College, College Park, Md.
McMurtrie, William, 100 William street, New York.
Macfarlane, T., Ottawa, Canada.
Magruder, E. W., Richmond, Va.
Mattern, L. W., Central High School, Washington, D. C.
May, D. W., U. S. Department of Agriculture, Washington, D. C.
Meng, James S., German Kali Works, New York, N. Y.
Miller, H. K., Agricultural Experiment Station, Lake City, Fla.
Mills, J. S., Washington, D. C.
Moore, C. C., U. S. Department of Agriculture, Washington, D. C.
Moore, J. F., Agricultural Experiment Station, Fayetteville, Ark.
Myers, J. A., 12 John street, New York, N. Y.
North, J., care Eimer & Amend, New York, N. Y.
Orton, W. A., U. S. Department of Agriculture, Washington, D. C.
Patrick, G. E., U. S. Department of Agriculture, Washington, D. C.
Patterson, B. C., Torrington, Conn.
Patterson, H. J., Agricultural Experiment Station, College Park, Md.
Penny, C. L., Agricultural Experiment Station, Newark, Del.
Perkins, W. R., Agricultural Experiment Station, Agricultural College, Miss.
Plunkett, W., 12 John street, New York, N. Y.
Price, T. M., Agricultural Experiment Station, College Park, Md.
Rand, Stephen, Washington, D. C.
Robb, J. B., Maryland Agricultural College, College Park, Md.
Robertson, A., Laidlaw Meekill Company, Richmond, Va.
Runyan, E. G., U. S. Department of Agriculture, Washington, D. C.
Sawyer, H. E., 620 Atlantic avenue, Boston, Mass.
Schlichting, E., U. S. Department of Agriculture, Washington, D. C.
Seidell, Atherton, U. S. Department of Agriculture, Washington, D. C.
Shiver, F. S., Agricultural Experiment Station, Clemson College, S. C.
Shutt, F., Ottawa Experiment Station, Ottawa, Canada.
Shuttleworth, A. E., Ontario Agricultural College, Guelph, Canada.
Smith, G. W., 75 West 45th street, New York, N. Y.
Spencer, G. L., U. S. Department of Agriculture, Washington, D. C.
Straughn, M. N., Maryland Agricultural College, College Park, Md.
Street, J. P., Agricultural Experiment Station, New Brunswick, N. J.
Stubbs, J. E., Agricultural Experiment Station, Reno, Nev.
Tillinghast, J. A., Agricultural Experiment Station, Kingston, R. I.
Tolman, L. M., U. S. Department of Agriculture, Washington, D. C.
Traphagen, F. W., Agricultural Experiment Station, Bozeman, Mont.
Trescot, T. C., U. S. Department of Agriculture, Washington, D. C.
Van Slyke, L. L., Agricultural Experiment Station, Geneva, N. Y.
Veitch, F. P., U. S. Department of Agriculture, Washington, D. C.
Voorhees, L. A., Agricultural Experiment Station, New Brunswick, N. J.
Wheeler, H. J., Agricultural Experiment Station, Kingston, R. I.

Wiley, H. W., U. S. Department of Agriculture, Washington, D. C.
Williams, C. B., Department of Agriculture, Raleigh, N. C.
Winton, A. L., Agricultural Experiment Station, New Haven, Conn.
Withers, W. A., Agricultural Experiment Station, Raleigh, N. C.

ORDER OF BUSINESS.

The following order of business was adopted:

The president's address.

Reports of the referees in the following order:

First. Report on nitrogen.

Second. Report on potash.

Third. Report on phosphoric acid.

Fourth. Report on soils.

Fifth. Report on ash.

Sixth. Report on foods and feeding stuffs.

Seventh. Report on liquor and food adulteration.

Eighth. Report on dairy products.

Ninth. Report on sugar.

Tenth. Report on tannin.

Eleventh. Report on insecticides.

Twelfth. Reports of special committees (abstract committee, food standards, fertilizer legislation, volumetric standards).

The president announced to the association that a representative of the National Grange was present and would address the members. He then introduced Mr. J. A. Tillinghast, of Kingston, R. I., who stated that he had come to invite the members of the Association of Official Agricultural Chemists to meet in connection with the National Grange at 11 o'clock at the National Hotel.

It was moved and carried that after the reading of the president's address the association should adjourn to meet with the National Grange at the National Hotel, and that it should reassemble in the lecture hall of the Columbian University at 1 o'clock p. m.

The president appointed Mr. Frear and Mr. Van Slyke a committee to wait upon the Secretary and Assistant Secretary of Agriculture and notify them that the members of the association would be pleased to have them visit the meeting.

Mr. Kilgore then addressed the convention as follows:

PRESIDENT'S ADDRESS.

GENTLEMEN OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS: In casting about for a subject on which to address some remarks to those attending the seventeenth annual convention of the association over which you have done me the great honor, which I highly appreciate, of calling me to preside, I have read with interest and profit all of the accessible former presidential addresses before this body and have found that without enactment on the part of the association, custom of sixteen years' standing has practically fixed the basis of the presiding officer's remarks, unless he essays to be a reformer. Without exception, these addresses have been short and have dealt with some phase of our history as an association, with present work or problems confronting the agricultural chemist, with specific discussion relating to the improvement of some of our methods for the analysis of agricultural products, or with suggestions for enlarging the field of our endeavor.

The records of the association show it to be one of action and business, and I shall endeavor to put what I have to say before you in a business way.

NATIONAL PURE-FOOD LAW.

Not only has the scope of our work been extended, but the influence of the association has grown. During the year we have been called upon as an organization to aid in the education of public sentiment and to bring influence to bear in favor of a national pure-food bill, with which we are all more or less familiar. Knowing that a number of the members of the association are actively engaged in the examination of food products and in aiding to execute State pure-food laws, and feeling that our entire membership is deeply interested in this important movement upon which so much of health and happiness for ourselves and those we represent depends, I used the name of the association and joined other scientific bodies in this country in asking our Congress to pass a national pure-food law. I trust that I may have the approval of the association in this action.

In North Carolina we are just preparing our first report on the examination of food products under our new pure-food law. We have been engaged in this work, as time permitted, for over a year and see the importance and necessity for a national law to help us when we can not help ourselves. As other scientific bodies have adopted resolutions on this subject, and in view of its importance, I suggest that this association, possibly through resolutions prepared by its standing committee on food standards, express itself fully in regard to the matter.

NATIONAL STANDARDIZING BUREAU.

Accurate and uniform standards are the basis of analytical and, in fact, all scientific work. Especially are they essential to the work of this association, the fundamental feature of whose organization is uniform methods as a means to uniform results. In the last Congress the prospects seemed fair for the passage of a bill establishing "The National Standardizing Bureau" under Government control. It would be the work of this bureau to investigate and adopt standards, and to provide facilities for testing and correcting the apparatus of various scientific and other workers, and to establish uniform systems. Since 1896 this association has had a standing committee on volumetric standards, and in the discussion and approval of the three reports made by this committee the association has shown its deep interest in the question of accurate and uniform standards. With this in view, I again used the name of the association in trying to help secure the passage of the bill to which reference has already been made.

I can not think of anything that is more essential to the progress of the work of this association than that involved in the question of proper standards; and, if I mistake not, it was here that this movement had its beginning in this country. The question was up for discussion in the 1896 meeting, when provision for a committee was made. That committee has made three reports of the results of its investigations, and a member of the committee brought the matter before the American Chemical Society, which is now active in its support of the measure. Our association and its members have, therefore, done their part of the work on this important question. Should we not now express fully and clearly our views on the subject of standards so that they may be used with the full force of the association in case the matter comes up again for agitation?

UNIFORM STATEMENT OF RESULTS OF FERTILIZER ANALYSES.

In addition to obtaining uniform, accurate, rapid, and inexpensive methods of analysis, this association has among its objects the securing of uniform modes of statement of results obtained by these methods. We have paid more attention in the past to getting proper methods, which was certainly the first and most important object to be attained, than to securing uniform statements of the results. The work of the committee appointed in 1897, and which reported in 1898, on the subject of uniform branding of fertilizer bags and uniform fertilizer laws, has done good in that it has been made the basis of an agreement and report of the State chemists of seven of the Southern States to the Association of Commissioners of Agriculture of the Cotton States. This association met in Raleigh, N. C., during the past summer, and after a great deal of discussion the chemists' recommendations were agreed to, and we feel that there is now a hopeful outlook for the plan going into execution. It is necessary for the chemists and commissioners of agriculture, or others having the business of the fertilizer controls in charge, to come together in these matters, and such has been our mode of attack in the South.

Not only was an agreement reached in this meeting as to the statement of analyses on fertilizers, but the committee was continued and enlarged and instructed to draft a uniform fertilizer law for the States represented in the association, and also to meet with the manufacturers if desirable and see if an agreement can not be reached in regard to the naming or misnaming of fertilizer brands, such, for instance, as "dissolved bone" and "dissolved bone and potash," when there is no bone present; and, further, to consider the character of materials entering into the different fertilizer brands, and to investigate the desirability of exacting the filing of a statement in regard to these, as is done in the case of the valuable constituents of a fertilizer.

I make this somewhat detailed statement to show the association that its actions have weight, and in the hope of inducing further discussion of this question in case the association has the time and inclination to do so. It may not at first thought seem so important to have uniformity in the naming of the valuable constituents of fertilizers, and so far as chemists alone are concerned it is not, for we understand the various synonymous terms employed in designating them, but when it is remembered that the burden of explaining these terms to the farmers and users of fertilizers devolves upon the chemist, the question of sameness and simplicity assumes weight, and it would be greatly to our advantage and credit if we called the same substances by the same names in all of the States of the Union. This association can bring this about, as it has in the case of methods, if it will only put its shoulder to the wheel, and we will then have accomplished another object set forth in our constitution.

INDIVIDUAL EFFORT AS A FACTOR IN THE GROWTH OF THE ASSOCIATION.

I now desire to make a few observations on facts as they have impressed themselves upon me in a study of the history of the growth of this association. The most prominent subject before the American public to-day is that of trusts, combinations, and organizations. So prominent have these become that it is generally assumed that the individual is swallowed up and lost sight of in the great aggregations. Is this really true? Certainly in union there is strength, but we have strength and influence, not only because of our organization but because care, conservatism, and strong individual efforts have directed the various phases of our endeavors in which much progress has been made. I feel that I am correct in the observation that no very important advance in the development of any of the methods of the association has been accomplished except where strong individual effort has preceded the submission of the questions to the general test of the association. Those who are familiar with the work of the association for a number of years know that with each of the subjects which have been under investigation are associated the names of a few of the workers of the association who have become prominently identified with the perfection of each particular process or method only because of having given a great deal of time, thought, and labor to the solution of the problems before them. It is not true that they deserve all the credit, nor is it right that they should receive it, for unless the association take up and subject their plans to the rigid test to which it submits everything, their ideas could not receive the acceptance and prominence which have been accorded them. Those men, however, because of their individual labors, deserve to be called captains of their causes.

We might trace the development of any of the older methods of the association, especially those employed in fertilizer analysis, to see how much is due to individual efforts in bringing any one of them to its present state of accuracy. The work on potash methods lends itself well to a study of this kind, and I shall refer briefly to the important steps in perfecting our methods for this determination.

The rather crude condition of the method generally used in 1880 and prior thereto may be imagined from the following description: "Solution in water; removal of sulphates, phosphates, and magnesia by barium hydrate; clearing with oxalate or carbonate of ammonia, and precipitating with platinum chlorid." The brevity of description was doubtless compensated for by the variation in manipulation. This method was used until 1884, when it was redescribed and adopted as the official method in such form as to require five filtrations, three evaporations, and two ignitions. By investigations conducted in 1885 one each of the filtrations, evaporations, and ignitions were omitted and the method of Lindo was given a test. This method, modified on the basis of the work and suggestions of Gladding, became one of our methods for potash determinations in 1886. As then described, only two changes need to be made to give us our most excellent Lindo-Gladding method of to-day. No change was made in this method from 1886 to 1888, when, on the basis of work done, especially at the New Jersey Experiment Station, it was directed to add ammonium oxalate to help remove lime. While a good many workers were now doubting the necessity of using sodium chlorid in this method, it remained for Winton to show, by a most carefully conducted series of experiments, the results of which were presented in a paper before the association in 1891, that it was not only not essential but was a hindrance and a great expense. This conclusion was verified by the general test of the association in 1892, when the term "sodium chlorid" was stricken from its place in the method.

Criticism, whether of individual work or of an association's position, is one of the strongest incentives to a thorough refutation of charges and the establishment of the correctness of the work or opinion beyond peradventure. Such criticism fortunately came upon our potash methods about 1893. Spurred on by the sting of unfavorable opinion, and under the wise and fortunate action of this body, which permits the long association of men with particular lines of work, our potash methods were subjected through the years 1894, 1895, 1896, and 1897 to such continuous, severe, and carefully planned and conducted tests on all classes of material that the results obtained must command the greatest respect and confidence of others, especially for our Lindo-Gladding method, and raise it in the estimation of its friends at home, where it was already most favorably known. While the association is to be congratulated and given credit as a whole for this excellent work, I feel that the greater part of the credit belongs to the two or three referees and associates whose individual efforts planned so well this continuous work and who verified personally the wisdom of their plans before submitting them to the general test of the association. To this I would add that we were greatly aided through the efforts of one of our members in securing this uniformly high opinion of our methods, by a better knowledge of the crystalline character of the end compound, the double chlorid of potassium and platinum, and of the conditions for obtaining it in a state best suited to accurate results.

So well has this work been done that the referee in 1896, in referring to the Lindo-Gladding method, said, "An experienced analyst, who follows strictly the instructions and uses a Gooch crucible, should find it rapid, economical, and accurate," while the referee for 1898 opened his report with these words: "The methods for estimating potash have been quite thoroughly studied by this association in years past, so far as potash salts and fertilizers are concerned," and he proceeded to test the applicability of the methods to different kinds of ashes. And, as further evidence of the satisfactory condition of our potash methods, it may be said that so excellent and ingenious a worker as Professor Ross could only suggest for investigation in 1899 the following question: "Which is better with which to saturate in making ignitions of such organic materials of cotton seed meal and tobacco stems, concentrated or 1:1 sulphuric acid?"

What I have said in regard to individual effort in connection with our work has been said without desire to detract from the great and supreme importance of the association, or to exalt unduly the work of individual members. I have merely stated some facts as they have impressed me in the study of our growth in the hope that they may induce more of our members to take up and study thoroughly some of the many phases of the subjects on which we are working and which are in such sore need of strong individual effort.

METHODS FOR THE EXAMINATION OF HUMAN FOODS.

In conclusion may I in a few words draw your attention to the growing prominence and importance of the examination of human food and drink. Recently a number of States have passed pure-food laws. We are in need of new methods and of concerted tests of old ones for the examination of food products. Our methods of fertilizer analysis are in very satisfactory condition, and this status has only been reached by dividing the subject into nitrogen, phosphoric acid, and potash, and giving to each detailed consideration. The food analyst is called upon to make not only the usual analysis of the various foods, condiments, and drinks, but also to examine them for adulterants, preservatives, and coloring matters. Would it not be well for the association to make a number of subdivisions of this subject, and place experienced men in charge of the investigation of each subdivision? The scope of our work would be somewhat enlarged by an action of this kind and the food chemists generally would no doubt attend our meetings more largely in the future than they have in the past if the work of the association were thus made of more interest and value to them.

APPOINTMENT OF COMMITTEES.

At the conclusion of the president's remarks he declared the seventeenth annual meeting of the Association of Official Agricultural Chemists open for business, and appointed committees as follows:

Committee on recommendations of referees: H. A. Huston, J. T. Anderson, C. H. Jones, F. W. Traphagen and the referee of the report under consideration.

Committee on nominations: A. L. Winton, J. P. Street, W. F. Hand.

Committee on resolutions: C. L. Penny, F. T. Shutt, H. K. Miller.

Committee on subdividing work on liquor and food adulteration: A. L. Winton, H. W. Wiley, A. E. Leech, William Frear, W. D. Bigelow.

The meeting was then adjourned in order that the members of the association might attend, in a body, the meeting of the National Grange.

FRIDAY—AFTERNOON SESSION.

President Kilgore called the meeting to order at 1 p. m.

The referee on nitrogen not being present, the report on potash was called for and was presented by the secretary as follows:

REPORT ON POTASH.

By L. S. Munson, *Referee*, and C. L. Hare, *Associate Referee.*

The potash work of the association for several years past has been confined largely to investigations of analytical methods. This year it was thought desirable to include in the work the investigation of such problems as have presented themselves in connection with the application of the present methods to fertilizer work. Accordingly, on July 16, three samples were prepared and sent out to 19 persons who had expressed a desire to cooperate in the work. Of these, 8 have reported results obtained by 11 different analysts, 6 have reported inability to do the work, owing to lack of time, and 5 have made no report whatever.

Sample No. 1 was cotton-seed meal.

Sample No. 2 consisted of a mixture of 162 parts of acid phosphate, 64 parts of ground tobacco stems, 10 parts of potassium chlorid, and 4 parts of magnesium sulphate.

Sample No. 3 was of 2 parts, No. 3A being acid phosphate and No. 3B chemically pure KCl.

The instructions sent out with these samples were as follows:

(1) Determine moisture in samples Nos. 1 and 2.

(2) Determine both water soluble and total potash in samples Nos. 1 and 2.

(3) Make mixtures of No. 3A and 3B as follows:

(*a*) Nine grams acid phosphate and 1 gram chemically pure KCl.

(*b*) Nine and five-tenths grams acid phosphate and 0.5 gram chemically pure KCl.

(4) Make blank determinations of potash in acid phosphate used in No. 3 and in reagents and determine potash in chemically pure KCl.

(5) Make all determinations according to official methods.

Samples Nos. 1 and 2 were for the purpose of testing the application of the method for determining water-soluble potash to such fertilizers as are made up entirely or in part of organic material. The object of sample No. 3 was to ascertain whether in mixtures of acid phosphate and potash salts it was possible to obtain the theoretical amount of potash added.

The following results were obtained:

TABLE I.—*Comparison between water soluble and total potash in samples Nos. 1 and 2.*

Name.	Address.	Sample No. 1.			Sample No. 2.		
		Moisture.	Water soluble K_2O.	Total K_2O.	Moisture.	Water soluble K_2O.	Total K_2O.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
B. F. Robertson	Clemson College, S. C.	9.17	1.68	1.80	6.44	5.20	5.46
C. C. McDonnell	do	9.11	1.63	1.77	6.39	5.16	5.49
C. L. Hare	Auburn, Ala	7.65	1.79	1.95	6.60	5.37	....
J. Q. Burton	do	....	1.84	1.92	....	5.40	5.46
A. W. Blair	Lake City, Fla	9.97	1.58	1.79	8.15	5.22	5.34
L. S. Munson	Washington, D. C	8.24	1.78	1.89	6.54	5.28	5.38
C. D. Holley	Orono, Me	9.64	1.58	1.75	6.50	5.03	5.45
F. B. Carpenter	Richmond, Va	....	1.51	1.75	....	5.06	5.33
S. H. Sheib	do	9.14	1.37	1.75	6.38	5.07	5.26
T. R. Gough	Agricultural College, Md.	7.53	1.65	[1]2.22	6.39	5.19	....
I. D. Sessions	Agricultural College, Miss.	8.26	1.44	....	6.26	5.12	....
Average		8.75	1.62	1.82	6.63	5.19	5.40

[1] Not included in average.

TABLE II.—*Results of analysis of mixture of acid phosphate and potassium chlorid.*

Name.	Address.	K_2O in C. P. KCl.	K_2O in acid phosphate.	K_2O in mixture of 9 gr. acid phosphate and 1 gr. KCl.	K_2O in mixture of 9.5 gr. acid phosphate and 0.5 gr. KCl.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
B. F. Robertson	Clemson College, S. C	62.62	0.04	6.01	2.94
C. C. McDonnell	do	62.61	.03	5.99	2.95
C. L. Hare	Auburn, Ala	63.20		6.13	3.11
J. Q. Burton	do			6.20	3.16
A. W. Blair	Lake City, Fla	62.96	.103	6.14	3.08
L. S. Munson	Washington, D. C	63.08	.155	6.21	3.14
C. D. Holley	Orono, Me	63.00		6.30	3.15
F. B. Carpenter	Richmond, Va		.06	6.19	3.10
S. H. Sheib	do	63.13	.08	6.09	3.09
T. R. Gough	Agricultural College, Md	63.30	.14	6.13	3.08
I. D. Sessions	Agricultural College, Miss	[1]60.65		5.89	2.92
Average		62.99	.083	6.12	3.07

[1] Not included in average.

Theoretical per cent of K_2O in mixture of 9 gr. acid phosphate and 1 gr. KCl 6.37
Theorctical per cent of K_2O in mixture of 9.5 gr. acid phosphate and 0.5 gr. KCl 3.23

From Table No. 1 it will be seen that the results obtained for water-soluble potash are extremely irregular. With sample No. 1 there is a variation of 0.47 per cent, with less than 2 per cent of potash present, and while the results obtained on sample No. 2 are more regular, there is still a wide variation. The question also arises whether the water-soluble potash so nearly represents the available potash in this class of materials as in fertilizers made up entirely of mineral ingredients. Since the fertilizer laws of some States recognize only the water-soluble potash, while the laws of other States permit total potash to be determined, this matter becomes of considerable importance.

With sample No. 3, the mixture of 9 grams of acid phosphate and 1 gram KCl gave an average of only 6.12 per cent, while the theoretical amount, based upon the analysis of the acid phosphate and the chemically pure KCl, was 6.35 per cent. The mixture of 9.5 grams of acid phosphate and 0.5 gram KCl gave only 3.07 per cent, while the theoretical was 3.23 per cent. The time at our disposal would not permit of an investigation into the cause of these low results.

While it was impossible to obtain as comprehensive results as were desired, owing to the late date when the samples were sent out, yet the results reported are sufficient to justify a continuation of the work along these lines in order to adapt the methods to these classes of fertilizing materials.

Mr. Huston stated that he was somewhat interested in the matter of the loss of potash and cited some work of the Kentucky Agricultural Experiment Station of several years ago, in which it was found that the more certain materials were boiled the more concentrated they became, and the less potash there appeared to be. He said for that reason the method of boiling described on page 21 of Bulletin 46 had been abandoned in his laboratory and the one substituted was as follows: Put 10 grams of the material in a nitrogen flask, graduated to 500 cc; add 200 cc of distilled water and force steam through the fluid for 30 minutes. The steam is introduced by means of a small lead pipe reaching to the bottom of the flask. Mr. Huston stated that by the use of this method an increase in the volume of fluid was obtained as the solution went on and a higher percentage of potash secured.

Mr. Shutt. In connection with the question of potash, it may be of interest to the members to know that we have found a very satisfactory method for the determination of potash in soils as follows: The potash being in solution (in hydrochloric acid in case of total potash or in 1 per cent citric acid solution in case of available potash), ammonia is added until the liquid is decidedly alkaline. Without any filtration, about two grams of chemically pure barium carbonate is now added and the whole boiled for about half an hour and filtered. To the filtrate, a few cc of ammonium oxalate, carbonate, and hydrate are added. After heating the substance is allowed to stand. The precipitated carbonates and oxalate are filtered off, the filtrate evaporated to dryness, and the residue gently ignited until ammonia fumes cease. If the ignited residue does not readily dissolve to a clear solution with a few drops of water, it is again treated with a few cc of hydrate and carbonate of ammonia, warmed and filtered, evaporated to dryness and gently ignited. The potash is now precipitated by platinic chlorid after acidulation of the solution by hydrochloric acid.

The report on nitrogen was next called for. Mr. Shiver stated that, owing to continued bad health, it was impossible for him to take an active part in the work on nitrogen and so he turned over the whole matter to Mr. Perkins, the associate referee. The president then introduced Mr. Perkins, who read the report on nitrogen as follows:

REPORT ON NITROGEN.

By W. R. Perkins, *Associate Referee.*

I was notified by the president of this association, about the first of last March, that on account of ill health the referee, Mr. Shiver, would not be able to attend to the duties of the position. He requested me to take up the work for this season, and a similar request was made by Mr. Shiver about the same time.

Owing to the late date at which this order was received, and not knowing at what time the meeting would be held, the time at my disposal for becoming more familiar with the workings of the methods under consideration was very limited. Consequently, the instructions for work were in accord with Mr. Shiver's suggestions, being practically the same as for last year with regard to the determination of available organic nitrogen by the neutral permanganate and pepsin-hydrochloric acid methods.

Considerable difficulty was experienced in procuring the materials for use in the work, and I was unable to get samples of hoof and horn meal and leather in its various forms in any quantity, though a small amount of sole leather was obtained locally, and after much grating and grinding a sufficient quantity was prepared for three samples of the raw material, and a like number of a mixture containing leather. Samples of these were sent out to two chemists with the other samples.

Toward the latter part of March a letter was addressed to twenty-three chemists asking whether they desired to assist in the work or not. Favorable replies were received from eleven, and a set of samples was sent to each of them. The samples were as follows: No. 1, bat guano; No. 2, tankage; No. 3, ground bone; No. 4, fish; No. 5, dried blood; No. 6, cottonseed meal; No. 7, mixed fertilizer, containing 150 grams each of cotton-seed meal, fish, and dried blood, 200 grams of ground bone, and 925 grams of acid phosphate; No. 8, raw leather; No. 9, mixed fertilizer, containing 56 grams of raw leather, 50 grams each of dried blood and cotton-seed meal, and 300 grams of acid phosphate. All samples were passed through a millimeter sieve, thoroughly mixed, and the bottles filled from the same pile. The directions for work sent with the samples were as follows:

DIRECTIONS FOR THE A. O. A. C. WORK ON NITROGEN, 1900.

(1) Thoroughly remix the material for analysis before beginning the work.

(2) Determine total nitrogen by Kjeldahl method, using on No. 1 the method as modified for nitrates.

(3) Determine available nitrogen by neutral permanganate method (Street), as follows: Into a 600 cc Erlenmeyer flask weigh an amount of sample equivalent, approximately, to 0.075 gram of nitrogen. Digest this with 100 cc of permanganate of potash solution (16 grams of pure $KMnO_4$ to 1,000 cc of water) over a steam bath, or over water vigorously boiling, for thirty minutes. Shake thoroughly after ten minutes, and wash down sides of flask with a small quantity of same strength permanganate solution at same temperature of flask, using a fine-tipped wash bottle for this purpose. After this shake gently two or three times, and in case much of the substance adheres to side of flask, wash down with as little permanganate solution as possible. Remove from flask and add 150 cc of cold water, filter through a heavy folded filter, about 18 cm in diameter, and wash with cold water till total filtrate equals about 700 cc. Dry and determine nitrogen in residue by Kjeldahl method.

(4) If you have time, after completing the above, I would be pleased to have you determine available nitrogen by the pepsin-hydrochloric acid method, as follows:

Weigh 1 gram of sample into a 150 cc flask and add 100 cc of pepsin-hydrochloric acid solution. This solution is prepared by mixing 5 grams of Parke & Davis's pulverized pepsin (guaranteed to dissolve 3,000 times its weight of freshly coagulated egg albumen) in 1,000 cc of hydrochloric acid diluted to a strength of 1 per cent.

The flasks are then kept for twenty-four hours loosely corked in a water bath having a constant temperature of 40° C. At the end of the second, fifth, eighth, and eleventh hours 2 cc of a 10 per cent hydrochloric acid solution are added, shaking the flask thoroughly at the same time.

After twenty-four hours digestion, filter, wash four or five times, using 20 to 30 cc of water for each washing, and determine nitrogen by the Kjeldahl method in the washed and dried residue.

From the 11 laboratories to which samples were sent, partial or complete reports were received from 9, embracing the work of 11 chemists. The results of the work appear in the tables below:

TABLE I.—*Total nitrogen.*

Analyst.	Bat guano.	Tankage.	Ground bone.	Fish.	Dried blood.	Cotton-seed meal.	Mixed fertilizer No. 7.	Leather.	Mixed fertilizer No. 9.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
F. B. Carpenter, Virginia....	11.64	7.64	3.65	8.38	13.44	7.28	3.30	....	....
R. G. Newborn, Virginia	11.64	7.64	3.72	8.32	13.50	7.24	3.27	....	....
J. A. Bizzell, North Carolina.	11.74	7.71	3.61	8.36	13.72	7.35	3.33	....	....
	11.82	7.65	3.63	8.24	13.67	7.27	3.25	....	....
J. W. Ames, Ohio............	11.78	7.64	3.73	8.27	13.59	7.36	3.34	....	....
T. C. Trescot, Washington, D.C.	11.59	7.62	3.71	8.25	13.24	7.15	3.35	....	....
A.W. Ogden, Connecticut...	11.69	7.59	3.82	8.36	13.55	7.24	3.32	....	....
	11.73	7.60	3.84	8.36	13.57	7.24	3.34	....	....
O. W. Knight, Maine........	11.49	7.76	3.71	8.40	13.49	7.36	3.41	....	....
	11.59	7.70	3.77	8.34	13.57	7.36	3.41	....	....
	11.65	....	....	....	13.45	....	....	....	....
C. D. Holley, Maine	11.50	7.68	3.68	8.40	13.47	7.28	3.44	....	....
	11.43	7.68	3.70	8.36	13.41	7.28	3.36	....	....
	11.56	7.70	3.70	8.38	13.43	7.26	3.40	....	....
	....	7.64	3.74	8.38	13.43	....	3.38	....	....
E. B. Ferris, Mississippi	11.54	7.58	3.65	8.18	13.40	7.19	3.17	6.74	3.08
	11.54	7.52	3.65	8.18	13.42	7.18	3.19	....	....
	11.52	7.62	3.68	8.24	13.46	7.12	3.25	....	....
	....	7.53	3.70	8.24	13.36	7.11	....	....	....
J. M. McCandless, Georgia ..	11.67	7.61	3.72	8.30	13.57	7.25	3.20	6.83	3.20
W. R. Perkins, Mississippi...	11.62	7.58	3.73	8.32	13.47	7.21	3.21	6.71	3.14
	11.70	7.58	3.67	8.34	13.40	7.25	3.19	....	....
	11.70	7.55	3.66	8.26	13.46	7.15	3.21	....	....
	11.58	7.49	....	8.27	13.36	7.21	3.24	....	....
	11.57	....	....	....	13.67	7.21	3.24	....	....
	11.59	....	....	....	....	7.15	....	....	....
	11.60	....	....	....	....	....	....	....	....
	11.55	....	....	....	....	....	....	....	....
J. B. Robb, Maryland	11.56	7.44	3.82	8.24	13.40	7.22	3.37	....	....
	11.58	7.52	3.77	8.30	13.34	7.18	3.38	....	....
	11.68	7.50	3.83	8.30	13.48	7.24	3.45	....	....
J. P. Street, New Jersey	11.51	7.44	3.82	8.13	13.26	7.18	3.20	....	....
Average	11.61	7.60	3.72	8.30	13.46	7.23	3.30	6.76	3.14

TABLE II.—*Results on digestion of organic nitrogen with neutral permanganate of potash.*

Analyst.	Bat guano.	Tankage.	Ground bone.	Fish.	Dried blood.	Cottonseed meal.	Mixed fertilizer No. 7.	Leather.	Mixed fertilizer No. 9.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
J. A. Bizzell, North Carolina	2.96	0.91	1.32	0.97	0.86	0.70	1.26	....	....
	2.92	.90	1.27	.91	.71	.60	1.24	....	....
J. W. Ames, Ohio	2.66	1.03	1.31	1.02	.57	.99	1.63	....	....
T. C. Trescot, Washington, D. C.	2.92	2.15	1.17	.96	1.06	.92	.88	....	....
A. W. Ogden, Connecticut	2.48	.89	.93	.83	.52	.39	1.02	....	....
	2.52	.93	.99	.86	.62	.52	1.15	....	....
	....	....	1.05	....	.83	.60	1.17	....	....
	....	....	....	....	....	.82	1.38	....	....
O. W. Knight, Maine	2.71	.84	1.02	.87	.61	1.12	1.20	....	....
	2.83	.80	1.04	.94	.54	1.00	1.17	....	....
	....	.86	....	....	....	.96	1.19	....	....
C. D. Holley, Maine	2.48	1.02	1.02	.86	.58	1.36	1.14	....	....
	2.69	1.11	.99	.94	.60	1.38	1.18	....	....
	2.81	1.15	.97	.94	.63	1.42	1.17	....	....
	....	1.11	....	....	....	1.08	....	....	....
	....	1.17	....	....	....	1.06	....	....	....
E. B. Ferris, Mississippi	2.24	.73	1.06	.82	.67	.57	1.05	5.75	1.39
	....	.69	1.10	.87	.65	.52	1.07	5.80	1.45
	....	....	....	....	....	.47	1.14	....	1.42
	....	....	....	....	....	....	.96	....	1.44
W. R. Perkins, Mississippi	2.30	.88	.96	.76	.67	.61	.94	5.85	1.09
	2.34	.80	.89	.81	.83	.75	.87	5.82	1.21
	2.40	.74	.76	.75	.87	.49	.83	....	....
	2.56	.65	.81	....	.80	.49	....	....	....
	2.55	.98	.82	....	.75	.84	.78	....	....
	2.30	....	....	....	.75	.75	.94	....	....
	....	....	....	....	....	....	1.03	....	....
J. B. Robb, Maryland	2.64	.86	1.62	.75	.70	.72	2.00	....	....
	2.50	.81	1.56	.84	.84	.70	1.96	....	....
	2.63	.73	1.65	.81	.78	.59	1.92	....	....
J. P. Street, New Jersey	2.73	.77	1.18	.65	.52	.45	1.33	....	....
	2.66	.74	1.14	.74	.56	.45	1.25	....	....

TABLE III.—*Availability of organic nitrogen by the neutral permanganate method.*

Analyst.	Bat guano.	Tankage.	Ground bone.	Fish.	Dried blood.	Cottonseed meal.	Mixed fertilizer No. 7.	Leather.	Mixed fertilizer No. 9.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
J. A. Bizzell, North Carolina	74.87	88.15	63.53	88.31	93.72	90.42	63.33	....	....
	75.21	88.28	64.91	89.02	94.86	91.79	62.42	....	....
J. W. Ames, Ohio	77.41	86.51	64.87	87.66	95.80	86.41	[1]51.19	....	....
T. C. Trescot	74.80	[1]71.80	68.46	88.36	[1]91.99	87.12	[1]73.43	....	....
A. W. Ogden, Connecticut	78.82	88.29	75.71	90.07	96.16	[1]94.61	69.36	....	....
	78.48	87.76	74.15	89.47	95.42	92.80	65.46	....	....
	....	....	72.58	....	93.88	91.71	64.86	....	....
	....	....	....	....	....	88.53	58.55	....	....
O. W. Knight	76.59	89.20	72.72	89.60	95.48	84.78	64.80	....	....
	75.55	89.71	72.19	88.76	96.00	86.41	65.68	....	....
	....	88.94	....	....	....	86.95	65.10	....	....
C. D. Holley, Maine	78.43	86.70	72.43	89.71	95.68	[1]81.31	66.47	....	....
	76.61	85.52	73.24	88.75	95.53	[1]81.04	65.29	....	....
	75.56	85.00	73.78	88.75	95.30	[1]80.49	65.58	....	....
	....	85.52	....	....	....	85.16	....	....	....
	....	84.74	....	....	....	85.43	....	....	....
E. B. Ferris	80.57	90.34	71.13	90.01	95.00	92.02	67.18	14.56	54.87
	....	90.87	70.02	89.40	95.15	92.72	66.56	13.82	52.92
	....	....	....	....	....	93.42	61.25	....	53.89
	....	....	....	....	....	91.52	....	....	53.24
W. R. Perkins	80.20	88.34	74.05	90.83	95.02	....	[1]70.71	12.55	61.34
	79.86	89.39	75.92	90.22	93.83	89.59	[1]72.89	13.00	65.17
	79.34	90.06	[1]79.45	90.95	93.54	93.19	[1]74.14	....	....
	77.96	91.24	[1]79.61	....	94.06	93.19	[1]73.52	....	....
	78.05	87.02	[1]78.10	....	95.38	88.33	[1]75.69	....	....
	80.20	....	[1]77.81	....	94.43	90.41	67.91	....	....
J. M. McCandless	79.70	89.22	74.83	90.72	94.32	90.90	66.89	13.76	49.37
	77.12	87.38	68.01	87.03	94.03	86.20	[1]55.31	13.17	43.75
J. B. Robb	77.24	88.50	[1]57.39	91.18	94.78	90.02	[1]41.17	....	....
	78.10	89.17	[1]58.94	89.85	93.73	90.30	[1]42.35	....	....
	77.32	90.24	[1]56.57	90.21	94.18	91.82	[1]43.53	....	....
J. P. Street	76.28	89.65	69.10	92.00	96.07	93.73	58.43	....	....
	76.88	90.05	70.15	90.89	96.22	93.73	60.93	....	....
Average	77.07	88.39	71.59	89.20	94.97	89.95	64.50	13.47	54.32

[1] Omitted from average.

TABLE IV.—*Results on digestion of organic nitrogen with pepsin-hydrochloric acid.*

Analyst.	Bat guano.	Tankage.	Ground bone.	Fish.	Dried blood.	Cottonseed meal.	Mixed fertilizer No. 7.	Leather.	Mixed fertilizer No. 9.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
J. A. Bizzell	5.14	3.45	.28	2.64	.64	.53	.51	....	....
	....	3.25	.29	2.80	.62	.58	.54	....	....
T. C. Trescot	5.86	4.20	.25	3.20	.61	.55	.58	....	....
A. W. Ogden	6.00	3.80	.19	2.78	.52	.48	.46	....	....
	6.06	3.84	.20	2.78	.52	.48	.48	....	....
W. R. Perkins	6.34	5.08	.36	3.76	.88	.79	.64	6.38	.92
	6.44	5.05	.32	4.13	....	.79	....	....	....
	....	4.74	....	3.61	....	....	....	....	....

TABLE V.—*Availability of organic nitrogen by pepsin-hydrochloric acid method.*

Analyst.	Bat guano.	Tankage.	Ground bone.	Fish.	Dried blood.	Cotton-seed meal.	Mixed fertilizer No. 7.	Leather.	Mixed fertilizer No. 9.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
J. A. Bizzell	[1]56.36	56.38	92.26	67.23	95.39	92.47	84.19	....	....
T. C. Trescot	49.43	44.88	93.26	61.21	95.39	92.30	82.65	....	....
A. W. Ogden	48.51	49.74	94.78	66.75	96.17	93.37	82.53	....	....
J. M. McCandless	49.33	....	92.47	70.12	96.31	92.27	81.56	14.49	73.75
W. R. Perkins	45.44	[1]32.71	90.27	[1]52.50	43.47	89.00	80.12	4.92	70.70
	[1]44.58	[1]33.10	91.68	56.45	....	89.00	....	....	....
	....	....	....	....	....	91.34	....	....	....
Average	48.18	50.33	92.45	66.32	95.34	91.39	82.19	9.70	72.22

[1] Omitted from average.

NOTES BY THE ANALYSTS.

O. W. Knight, Orono, Me.—Found it impossible to remove residue completely from flasks after digestion with K_1MnO_4 solution, so 500 cc breakers were used instead for the majority of the samples.

C. D. Holley, Orono, Me.—Used 500 cc round-bottom flasks placed well down in steam bath and surrounded by live steam. Found it impossible to wash down sides of flasks thoroughly. Used 500 cc breakers with part of the work, stirring instead of shaking.

T. C. Trescot, Department of Agriculture.—With sample No. 6 the permanganate was entirely reduced before the expiration of 30 minutes.

J. P. Street, New Brunswick, N. J.—Made the digestion over a steam bath, and followed the instructions strictly in every respect. Thinks the addition of water to the flask before filtering a drawback on account of its retarding the determinations. Mr. Street also sends results of his work with thirty classes of organic materials by this method. Quite a number of determinations on bone, fish, tankage, blood, and leather are given and show very satisfactory results.

A. W. Ogden, New Haven, Conn.—Directions were followed closely on the permanganate, but on the pepsin-hydrochloric acid method the solution was made by dissolving 5 grams of Parke, Davis & Co.'s 1:3,000 pepsin in 1,000 cc of hydrochloric acid diluted to a strength of 0.2 per cent. The Connecticut station has always used this strength solution. Mr. Ogden gives a number of comparative analyses by the permanganate and pepsin-hydrochloric acid methods on bone, fish, tankage, blood, leather, cotton-seed meal, and castor pomace, which showed about the same relative figures as did the light materials in the official samples, except on bone. With this the average of two analyses was higher by the permanganate than by the pepsin-hydrochloric acid method. He also made duplicate determinations on five mixed fertilizers. These gave the availability of the nitrogen by the permanganate method from 25 per cent to 40 per cent higher than by the pepsin-hydrochloric acid method. The materials in both instances were washed with 300 cc of water before treatment with the solution.

DISCUSSION OF RESULTS.

The directions for work on total nitrogen were followed, so far as reported, by all of the analysts taking part in the work except Mr. Trescot, who used the Gunning method. There is considerable difference between the highest and lowest results with every sample in Table I, being between 0.2 and 0.3 per cent on all except the bat guano and dried blood and 0.39 per cent and 0.48 per cent respectively for them. With the care taken in mixing the materials these variations are difficult to account for from this cause alone, though we recognize the fact that a lack of uniformity in samples may always be looked for as a possible source of error in comparing work of this kind. It is noticeable from the tables that there is a tendency throughout in the results of some of the analysts to be either high or low on all of the samples.

Rejecting a few of the highest and lowest results in Table II, the bat guano, bone, fish, dried blood, the two mixed fertilizers, and the leather, appear to agree quite closely, while with the tankage and cotton-seed meal the figures are more variable. The analysts, however, with few exceptions, got very concordant results on their different determinations, indicating that under the same conditions the method is very reliable.

In the digestion of the samples as followed in this work, the cotton seed meal completely destroys the color of the permanganate before the expiration of the thirty minutes and the tankage approaches very near to that result. Under these conditions it is very evident that any variation in the quantity of solution used will be attended by some change in the results. A lack of accuracy in measuring the prescribed quantity or differences in the amount of solution used in washing down sides of flasks might account for some of the wide variations in this table. The suggestion as to washing down materials adhering to flasks was made after doing some work on a sample of bone meal, with which changing the quantity of permanganate solution had very little effect on the quantity of nitrogen undissolved, even though as much as 200 cc were used.

The figures in Table III are derived by computation from those of Tables I and II. They show the determined availability as calculated from the average of each analyst's results on total nitrogen, and the separate determinations of that element left undissolved. The results, unlike those in the preceding table, show the greatest variations with the bone and two mixed fertilizers, all low nitrogen. The results with bat guano, tankage, fish, dried blood, cotton-seed meal, and leather, omitting five results, show to much better advantage. In expressing these results in "per cent of nitrogen available," the differences are very much magnified with samples of low nitrogen content, and results on such materials must agree within very narrow limits to be of any value. A difference of 0.2 per cent of undissolved nitrogen in blood would mean about 1.5 per cent in its availability. Such a difference in results on bone, or the mixed fertilizers above, would mean fully 6 per cent when calculated to per cent of nitrogen available, and in samples containing 2 per cent of nitrogen the difference would be 10 per cent.

The mixed sample No. 7 was made of the same raw material as that distributed for this work. Using the averages on these materials given in Tables I and II, its computed analysis should be:

	Per cent.
Total nitrogen	3.23
Availability of nitrogen	87.27

As determined by the different analysts it was:

	Per cent.
Total nitrogen	3.30
Availability of nitrogen	64.50

Mixed fertilizer No. 9, in like manner contained, as computed:

	Per cent.
Total nitrogen	3.10
Availability of nitrogen	72.00

As determined:

	Per cent.
Total nitrogen	3.14
Availability of nitrogen	54.32

The difference in the determined availability of these two samples as compared with their computed availability is very great, and was undoubtedly due to the presence of soluble phosphoric acid.

To test the effect of the presence of this substance on the results of some of these samples by this method, two sets of determinations were made. The same quantity of material as used in the regular work was weighed out, and from 2 to 3 grams of acid phosphate added to each, otherwise they were treated as in the official work. The amount of nitrogen in residues after digesting and washing was:

Tankage.	Bone.	Fish.	Blood.	Cotton-seed meal.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
2.67	1.91	4.71	10.50	1.41
2.80	1.98	....	10.23	1.86

It was noticed in treating the raw materials that the filtrate from the residues after digestion with permanganate solution gave a precipitate with a water solution of soluble phosphate. This probably explains the high results just given, as well as the unsatisfactory results with the mixed fertilizers of the tables.

As bearing on this point, the following results would indicate the necessity of washing the samples of mixed fertilizers before treatment with permanganate solution:

TABLE VI.—*Results on organic nitrogen in mixed fertilizers by the neutral permanganate method after washing sample.*

Analyst.	Material.	Amount of material used.	Availability.
		Grams.	*Per cent.*
J. P. Street	Mixed fertilizers	2.34	78.50
J. P. Street	.....do	2.34	77.84
A. W. Ogden	.....do	2.00	82.00
W. R. Perkins	.....do	2.30	87.27

While these results do not agree as closely as could be desired, they approach much nearer to the theoretical figures than do those of this sample in Table III.

A glance at the few results in Tables IV and V will show that with the pepsin-hydrochloric-acid method the results on bone, dried blood, cotton-seed meal, and the mixed fertilizers are very close, but with the bat guano, tankage, and fish they do not agree at all, even after omitting the very low results of my own work on these samples.

In conclusion, it must be said that the work for this year has not resulted in any material advance toward solving the problem under consideration, and I very seriously doubt if either the neutral permanganate or pepsin-hydrochloric acid method can be so perfected as to render them of much value in detecting adulterations in the nitrogen supply of fertilizers. Twenty-six per cent of the nitrogen in sample No. 9, for instance, is supplied in raw leather, so great an amount that the presence of the material is unmistakable, yet the availability of the nitrogen in the mixture, as determined in the raw material by the permanganate and pepsin-hydrochloric acid methods, is 72 per cent by the former and 70.6 per cent by the latter, considerably higher than bat guano, tankage, or fish by the pepsin method and higher than bone by the permanganate method. With less leather, as would undoubtedly be the practice of fertilizer manufacturers using it, the results would be still higher, and, consequently, there would be more confusion.

I believe, however, that the permanganate method, to which my work has been largely confined, can be made to give good results in the hands of different analysts by carefully conducting the work with reference to the following points:

(1) Weigh out the same amount of material and wash thoroughly in case of mixed fertilizers.
(2) Use the same quantity of solution, not washing down sides of flasks.
(3) Keep a uniform temperature.
(4) Bring all of the material under the action of the solution for the full time.
(5) Avoid the passing of the finer particles of the substance through the filter in washing.

I would suggest that the succeeding referee give the above points his consideration in any further work on this method, and recommend that the neutral permanganate method (Street) and the alkaline permanganate method (Jones) be referred to the referee for 1901.

The president then introduced Mr. Dyer, of London, England, who addressed the association as follows:

ADDRESS OF MR. DYER, OF LONDON, ENGLAND.

Gentlemen of the Association: I thank you for this kindly greeting. It is a great pleasure to me and a privilege to meet you all here, because I have followed closely and with much interest during the last half dozen years the work you have been doing on this side of the water, in connection, more especially, with soil work. For a considerable length of time I have tried to make some advancement in our methods of determining the available constituents in soils. I published a paper on the subject, and that paper at once met with interest on every hand on this side of the Atlantic. You at once set to work here to use my methods and to try them comparatively with other methods on parallel lines. My own work, which has been on availability, has been almost entirely confined to mineral constituents. This question of available nitrogen, which is occupying the attention of the meeting at the present moment, is one to whose discussion I am afraid I can not offer any useful contribution, but as your president has been so kind as to call upon me, I can not let this opportunity pass of expressing my great pleasure, the great delight it has been to me to see you taking up my little bit of work as you have here and doing so much which I hope will in the end prove to be the means of improving methods and making an advance upon the steps which I have endeavored to make.

I am in America at the present time as the representative of the Rothamsted Agricultural Experiment Station, with which I am not officially connected, but where I have done a great deal of work, and I am here to deliver the Lawes lectures. I may say that in the Rothamsted laboratory considerable interest has been taken by Sir Henry

Gilbert in the papers of Professor Hilgard and his associates on the determination of available nitrogen in soils in the work which has been done by many of you on the lines sketched out by Hilgard. Large numbers of Rothamsted soils have been subjected in the laboratory to investigation by Hilgard's method, or rather by the association's modification of what is generally known as the Hilgard method. I am afraid I can not say that the results in the investigation of the soils which we have at Rothamsted promise to give us very much light. We find that the proportion of nitrogen soluble in dilute alkali seems to bear a pretty constant relation to the total nitrogen in the soil, and the relationship between the nitrogen and the soluble organic matter does not appear to throw any definite light upon the comparative availability of the nitrogen. We do not get very much more information than would be given from the total nitrogen, but that is, of course, comparing a number of different soils, originally of the same type, that have been modified by the process of cropping and manuring.

I thought you would like to know that as you take an interest in methods which we sometimes originate on our side, so we at the same time follow your work and interest ourselves in the methods you put forward. I thought you would like to know that the association's method for determining nitrogen in soils had been subjected to examination by us.

The President. The association is very glad to hear from Mr. Dyer and we are glad to have him join in the discussion of our reports and papers.

Mr. Withers then presented the following paper:

THE RATE OF NITRIFICATION OF CERTAIN FERTILIZERS.

By W. A. Withers and G. S. Fraps.

The value of any fertilizer depends on its availability to the plant, that is, the readiness with which it can be taken up directly by the plant or converted into forms which can be assimilated. Nitrogen can be assimilated directly by ordinary agricultural plants in the form of certain organic compounds, ammonium salts, and nitrates, nitrates being the preferable form. Different plants behave differently toward these forms; some are not benefited by ammonium salts when young, and others are; some feed upon nitrates during their entire period of growth, and others are not benefited by them until they have attained some size. Nitrates, however, appear generally to be most valuable.

When combined nitrogen, in whatever form of combination, is placed in the soil, it is converted into ammonium salts, nitrites, and finally nitrates, with greater or less speed, depending on the form of combination, the temperature, condition of the soil, etc., provided certain living organisms are present, and they generally are. If in any given soil we determine the relative rate with which nitrogenous fertilizers, which can not be utilized directly by the plant, are converted into nitrates, it should throw some light upon the relative values of these particular fertilizers to plants.

Müntz and Girard (Central-Blatt f. agr. Chem.: 20, 656 (1891) abs.) have determined the rate of nitrification of certain fertilizers. A small quantity of the fertilizer was intimately mixed with natural soil, and kept at the temperature of 15°–25° C., properly moistened, and at the end of a given period leached with water, and the nitrate determined in the extract. The nitrate existing in the soil at the beginning of the experiment was previously determined. The time was thirty, thirty-two, and thirty-nine days for different sets. The nitrogen converted into nitrates and the nitrogen recovered from the soil by horse-tooth corn in two years is shown in the following tables:

Percentage of nitrogen oxidized and amount thereof recovered by the crop of two sucessive years.

Fertilizer.	Nitrified in 30 days.	Recovered by corn.
	Per cent.	*Per cent.*
Ammonium sulphate	75.0	76.7
Dried blood	72.4	55.0
Roasted horn, fine	71.0	60.1
Flesh meal	70.4	....
Horn trimmings, fine	55.5	53.3
Poudrette, rather coarse	18.1	18.1
Roasted leather, fine	11.6	38.3
Leather chips, raw	0.39	....

In another series the order of nitrification was as follows, the substance nitrified to the greatest extent being given first: Bat guano, dried grasshopper, dried cockchafers, flesh meal, dried blood. There is very little difference between the three substances named last. The nature of the soil had a great influence on the change. Nitrification was most active in a light soil from Jourville, used in the experiment referred to above, next in a garden soil, then in a chalky soil, then in a marled moor soil. Very little nitrification occurred in a very calcerous clay except with cow manure and yellow lupines, which loosened its texture, and none in an acid moor soil, with the same two exceptions.

P. Bonâme (Exp. Sta. Record: 9, 732, abs.) determined the nitrates in the drainage water from a sandy soil deficient in lime to which fertilizers had been added. The order of nitrification at the end of the first month was found to be—fish guano (most rapid), blood, fertilizer, oil cake, and ammonium sulphate. When calcium carbonate was added nitrification took place more rapidly, but the order was still dried blood, oil cake, ammonium sulphate.

In the experiments performed by us the fertilizing materials were those sent out by the referee of the association for this year to test the methods for determining the availability of nitrogen. A quantity of material equivalent to 0.6 gram of nitrogen was intimately mixed with 1,000 grams of soil, the sample being sifted through a coarse sieve, 6 meshes to the inch. The soil was then placed in precipitating jars and kept in a dark closet, adding enough water to make the percentage 11.6. At suitable periods three of the jars were weighed, and the loss of water replaced. The temperature was 28°–30° C., and the time was 3 weeks. When calcium carbonate was added the amount was just sufficient to combine with the nitrogen of the fertilizer if the entire amount were converted to nitric acid. At the end of the experiment the nitrates were leached out, and the amount determined by the Tiemann-Schulze method. The amount of nitrates in a blank experiment was deducted from the total.

On account of the small percentage of ammonium sulphate nitrified in the first series, the experiments with cotton-seed meal and ammonium sulphate were repeated, the time being twenty-six days, the temperature 23°–26° C., and the sample being moistened as before. The soil was taken from the same field as in the first series, but differed from it somewhat, as is shown by the fact that it contained 0.1641 gram of nitrogen per kilogram as nitric acid, whereas the former sample contained only 0.0595 gram.

As to the availability of nitrogen, we have selected the results obtained by vegetation tests with oats and Hungarian grass by Jenkins and Britton (Connecticut State Station Report, 1897, p. 257) and in the laboratory of the North Carolina station with the same materials nitrified by Bizzell, using the pepsin-hydrochloric acid and the neutral permanganate methods. These results are shown in the following table:

Comparison of the results obtained by vegetation tests, pepsin hydrochloric acid and neutral permanganate methods in determining the availability of nitrogen.

Fertilizer.	Rate of nitrification (time, 3 weeks).				Availability.		
	Without $CaCO_3$.		With $CaCO_3$.		Soluble in $KMnO_4$.	Soluble Pepsin-HCl.	Vegetation tests.
	Per cent.	Rank.	Per cent.	Rank.			
					Per cent.	*Per cent.*	*Per cent.*
Dried blood	34.8	100	54.9	100	94.4	94.7	73.3
Cotton-seed meal	33.9	97	54.8	100	91.1	91.1	64.8
Second series	26.7	....	....	....			
Dried fish	30.3	87	46.5	85	88.7	67.3	63.9
Tankage	26.2	75	34.8	63	88.3	56.4	49.4
Bat guano	22.4	64	35.8	65	75.1	56.4	....
Bone	18.9	54	16.6	30	64.2	92.3	16.7
Ammonium sulphate	1.3	4	31.1	55	100.0	100.0	....
Second series	3.4	....	32.6	....			
Nitrate of soda	....	....	....	....	100.0	100.0	100.0

It will be seen by an examination of the table that there is a very close correspondence between the order representing the rate of nitrification during three weeks and that representing the availability as shown by chemical and vegetation tests, except in the case of sulphate of ammonia. It seems to be nitrified much more slowly than the organic compounds used, and yet in many experiments in the field it gives results closely approximating those given by nitrate of soda. These results give rise to some interesting questions in regard to the availability of nitrogen in its different forms and also in regard to nitrification. A more detailed account of this work will be found in Bulletin No. 176 of the North Carolina station. This investigation will be continued by us.

Mr. FREAR. What was the character of the soil with which you mixed these materials, as to basicity or acidity?

Mr. WITHERS. I do not think we made this test. It was a soil which had been under cultivation for some time, was not very rich, and not much organic matter in it.

Mr. FREAR. Was it rich in basic material?

Mr. WITHERS. No; it did not have much lime, and when we added lime to the soil, nitrification took place much more rapidly than in other circumstances. There was not an excess of lime in the soil.

Mr. Ewell. I would like to ask if you offer any reason for the low rate of nitrification of ammonia in this case?

Mr. Withers. I suppose I can not offer any explanation that would be conclusive, but one explanation that seems to be indicated by it is this: It is reasonably supposed that organic compounds change first their ammonia salt and then change the nitrate. It would seem that the ammonia salt would undergo nitrification much more quickly than any form of organic compound. It may be that the difficult decomposition of the ammonium sulphate in the soil would account for the fact that the nitrification of the ammonia in ammonium sulphate would not take place as rapidly as nitrification of organic compounds.

Mr. Withers then cited the figures in another experiment along the same lines.

Mr. Ewell. What would be the concentration of the ammonia in the soil?

Mr. Withers. Six-tenths gram of nitrogen to 1,000 grams of soil. We had the same quantity of nitrogen in every case.

Mr. Frear. In this connection, if it is the ammonia which produces this effect, why would not the effect be intensified with an increased quantity of calcium carbonate? The effect might have been due to sulphuric acid which was set free. Of course acid will accumulate in proportion to the quantity of nitrogen which has been acted upon.

Mr. Ewell. I do not think there is any doubt about the acid.

Mr. Frear. The effect of ammonium sulphate fertilizers has been so thoroughly demonstrated that it need scarcely be mentioned. Our own experiments have been continued about twenty years on the same soil. We have found the nitrate ammonium sulphate plots to be very distinctly acid. Clover is crowded out and even the grass is pretty well crowded out, and the ordinary red sorrel has come to take its place. On examining these plots for nitrogen, I find we have a large excess of nitrogen. This may be due to the soil, but it may also be due to the failure of the ammonia to be nitrified.

Mr. Wheeler here remarked that the results obtained by Mr. Withers were so remarkable he would like to ask what kind of pots the experiments were conducted in.

After some further discussion on the subject the following paper was presented by Mr. Williams.

VARIABLE AMMONIA RESULTS IN MIXED FERTILIZERS.

By C. B. Williams.

As the determination of ammonia in commercial fertilizers is one of great importance on account of the high valuation of ammonia, it was thought that it would not be amiss to present at this meeting of the association some results obtained during the spring of 1899 in the laboratory of the fertilizer control of the North Carolina Experiment Station.

It has been, and still is, the custom of the control to prepare all fertilizers by grinding until they will pass through a sieve with perforations 1.25 millimeters (20 mesh) in diameter and then thoroughly mix with a spatula. Frequently it was found that on samples prepared in this way it was absolutely impossible to obtain concordant results, no matter how carefully the work was performed. It was not uncommon to find that duplicates weighed out at the same time and worked in exactly the same way gave results as divergent as 1 per cent. At first it was thought that these abnormal differences were due to leakages in the nitrogen distillation apparatus, but upon thorough inspection it was seen that this could not be the cause, as all connections were tight and secure. After many futile efforts to detect the cause of these great differences, it was finally observed that in every case the variations occurred on high grade complete fertilizers, whose ammonia was derived from either blood, fish scrap, tankage, or nitrate of soda.

Now, when such fertilizers are prepared by being forced through apertures 1.25 millimeters in diameter and then mixed, the relative coarseness of the materials furnishing nitrogen is so great as compared with the other components of the fertilizer as to make it absolutely impossible to obtain a homogeneous sample for analysis, even when the greatest care is exercised in mixing.

Further, it was found that the heterogeneity of the samples constantly increased after preparation, due to the tendency of these materials to separate from the others when the nitrogenized material used was either blood, fish scrap, or tankage. On account of their coarse mechanical condition and light specific gravity upon agitation, a constant concomitant of weighing, they will rise to the top of the sample. With these conditions, it is very easy to see that the ammonia results will vary directly with the quantity of this coarse nitrogenized material, weighed out for each determination. When a fertilizer bottle containing one of these fertilizers is tilted the coarse, light particles on top of the sample will roll to the lower side and the results will depend upon whether you take the portion for analysis from the upper or lower side of the tilted bottle. When this was noticed a portion of the coarse lower part and a portion of the fine upper part from almost all the samples were selected by the spatula and weighed separately and analyzed. By this selection it was not infrequent to obtain results differing by as much as 1 or 2 per cent of ammonia. In the subjoined table the results in the third column are from the fertilizers when they were prepared by being passed through a sieve with perforations 1.25 millimeters (No. 20) in diameter, while those in the second are samples that have passed a sieve with perforations 0.625 millimeter (No. 40) in diameter.

Fertilizer No.	Preparation.	
	Passed through sieve with perforations 0.625 mm. in diameter.	Passed through sieve with perforations 1.25 mm. in diameter.
	Per cent NH_3.	*Per cent* NH_3.
1077	5.15	5.68
1077	5.20	4.44
1077	5.22	4.76
1077		4.88
1077		4.76
1077		4.93
1077		4.78
1077		[1]5.40
1077		[2]4.55
1094	7.50	7.08
1094	7.48	6.96
1094	7.53	6.77
1094	7.58	6.81
1094		7.20
1094		7.89
1111	5.66	5.60
1111	5.61	5.79
1111	5.71	5.63
1111		5.37
1111		[2]5.55
1111		[1]6.01
1111		[1]6.90
1183	8.86	8.32
1183	8.81	[2]8.72
1183		[1]9.14
1183		[1]9.34
1183		8.18
1190	3.04	2.43
1190	3.04	2.63
1190	3.13	2.77
1190	3.06	[2]2.32
1190		[1]2.91
1366	8.69	9.12
1366	8.69	8.94
1366	8.64	8.75
1366		8.89
1366		8.84
1366		8.85
1366		8.91
1182	8.01	7.66
1182	7.98	[2]6.37
1182		[1]8.33
1182		[2]7.65
1182		[1]8.15
1258	2.84	2.66
1258	2.77	[2]2.96
1258		[1]3.08
1258		2.96
1277	3.08	2.74
1277	3.06	[2]3.30
1277	3.11	[1]4.10
1277	3.06	
1280	2.33	1.94
1280	2.21	[2]2.25
1280	2.19	[1]2.40 [1]2.96
1297	4.52	4.76
1297	4.42	4.65
1297		4.20
1297		4.83
1303	2.65	2.62
1303	2.70	2.78
1303		2.43
1303		2.50
1192	2.99	2.59
1192	3.01	[2]2.53
1192	3.01	[1]2.80
1222	3.88	3.56
1222	3.91	[1]4.59
1222	3.96	[2]3.40
1222	4.02	3.71
1227	2.74	2.55
1227	2.77	2.63
1227		[2]2.55
1227		[2]2.70
1227		[1]3.28
1236	3.08	3.06
1236	3.18	3.16
1236	3.20	[2]2.77
1236		[1]3.75
1254	5.15	4.88
1254	5.15	5.17
1254	5.22	[2]5.00
1254		[1]6.07
1324	2.77	2.50
1324	2.74	[1]3.05
1324	2.70	
1076		6.73
1076		6.51
1076		6.90
1076		6.22
1076		6.80
1076		6.29
1076		6.58
1081		8.72
1081		8.33
1081		8.72
1081		8.81
1180		6.07
1180		[2]6.17
1180		[1]6.85
1180		6.39
1180		[1]7.17

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[1] Selected with spatula, the coarse part for analysis.

[2] Selected with spatula, the fine part for analsvis.

Upon a study of the table it is seen that where the samples were forced through a No. 40 sieve the lowest difference was 0.02 per cent ammonia, and the highest was 0.14 per cent; while the results obtained when the samples were passed through a No. 20 sieve gave as the lowest difference 0.27 per cent, and the highest 1.96 per cent. In all these determinations the modified Kjeldahl method was used with 0.7 gram substance.

Even upon a superficial glance at the results in the table it is easy to see the necessity of at least reducing our fertilizers to a fine mechanical condition before attempting to make an analysis of them. There is not the slightest doubt in my mind that to reduce fertilizers containing fish scrap, blood, tankage, or nitrate of soda to a homogeneous state they must be forced through a sieve with apertures not much larger than those of the No. 40 sieve. It seems fair to assume that not only are the ammonia results affected by the mechanical condition of fertilizers as in the above, but also those of potash and phosphoric acid. It is hoped that during the present fertilizer season this work may be supplemented by that on more samples.

Mr. Myers. As some of you know, I have an interest in the nitrate of soda to the extent of promoting its use as much as possible, and I encounter this difficulty, which you gentlemen can correct if you will, and that is the manufacturers of commercial fertilizers complain that the State chemists fail to give them credit for nitrate of soda when mixed with other goods. The nitrate of soda, or any nitrate for that matter, is certainly the most available form of nitrogen, and I think the day is not far distant when the State chemists of this country and the chemists of other countries will find it to the interest of their inspection to state the nitrogen composition of a fertilizer just as you do the phosphoric-acid composition of a phosphate. Some of the stations are already doing it in this country, and I wish I could induce you to consider the advisability of presenting this composition of your fertilizers more clearly in your reports. I do not want to criticise in any way or ask for unnecessary favors, or anything of that kind, but this is based on scientific grounds, and where a manufacturer is using an available form of nitrogen, as in the case of a nitrate, he certainly should have credit for it.

I want to say further that there will be much more nitrate, probably, in this spring's fertilizers than in those of last spring, for the reason that, owing to the proficiency in handling the raw materials, nitrate is now probably the cheapest ammoniated material in the market, and it is very desirable that manufacturers should have credit for using it.

Mr. Withers. Mr. Wheeler has said, "These results are so at variance with those which are usually obtained." I would like to know to what results he has reference.

Mr. Wheeler. I intended to say that the results are so at variance with those we consider as the usual experience. I, perhaps, did not use those words, but that is what I intended to state.

I am about to indorse what the last speaker has said in regard to the nitrate of soda. The ground taken is strictly correct upon a scientific basis. In some States there may be pressure to complete the work in as short a space of time as possible, and, therefore, there are inducements to leave out determinations which should be made, not only in the interests of those who are selling good goods, but in the interest of the consumer. I think the association should express a sentiment in regard to this matter. I move that it is desirable that the form of nitrogen should be determined and reported on in the analysis of fertilizers.

Motion seconded and carried.

Mr. HUSTON. Two years ago a committee of experiment station chemists presented a report on uniform methods of standard fertilizer analysis. All forms of nitrogen were included in that report. Now, before we can arrive at any very satisfactory state of affairs in regard to this, we must do some work at home, in the various States. There is no State law existing for making a distinction between the forms of nitrogen. In my own State it is simply total nitrogen.

Mr. FREAR. In Pennsylvania we make only total nitrogen. There are so many fertilizers sold under different brands, I fear fewer analyses of the brands would be made than the farmers would want if we attempted to make three determinations of nitrogen instead of one.

Mr. Shutt then referred to some work done in his laboratory on soils containing different percentages of water and gave a description of the method in use for the determination of nitric and nitrite nitrogen in soils as follows:

> One hundred grams of the fresh undried soil is placed in a wide-mouth bottle and 1,000 cc of ammonia-free water added. The mixture is kept well shaken for one hour; it is then allowed to stand one hour. Two hundred and fifty cubic centimeters are now decanted and distilled and the distillates nesslerized in the usual manner and recorded as free ammonia. Another 250 cc is at the same time decanted and placed in contact with a zinc copper couple. After 24 hours this is distilled and the distillates nesslerized. From the results the amount of ammonia obtained the day previously is subtracted and the difference calculated to nitrogen present as nitrates and nitrites.

Mr. MCDONNELL. From the remarks of Mr. Myers, I do not quite understand what the trouble is in the case of the manufacturer. Is it that the chemist is not able to get the entire amount of nitrate of soda? In our experience there might be two sources of error; one the difficulty of getting the nitrate out of the sample by the Gunning or Kjeldahl method, as modified for nitrates, and another is that there is certainly sometimes a loss of nitrogen from the loss of nitrate in fertilizers due to the presence of acid phosphate. In regard to the other matters, nitrate of soda being cheaper, there would be no trouble about using plenty of nitrate.

Mr. MYERS. I suppose this calls for a little explanation. The claim of the manufacturers is that, in the first place, the methods of analysis do not distinguish between the forms of nitrogen and indicate the nitric nitrogen. In the next place it is admitted on all hands that if you mix nitrate of soda with a strictly acid phosphate, of course there will be a loss. That is a technical difficulty that can not be overcome, and I suppose will not be for some time. But the point we wish to make is, that we desire, as soon as it can be done without discommoding the State chemists any more than possible, we desire, I say, to get the chemists to state what brands of fertilizer have nitrate in them. It is not every year that nitrate is cheaper than other sources of nitrogen. This year it is due to the trust in ammoniating materials.

Mr. MACFARLANE. I would like to say in regard to this discussion that the fertilizer act in Canada obliges the manufacturer, when he sends in a standard sample every year for examination and analysis, to state the materials that have been used in its manufacture. This is published along with the results of analysis. We never make any statement as to what fertilizers should contain nitrates, but in a case where nitrates have been found, by the test which this gentleman has spoken of, it is mentioned.

Mr. FREAR. This matter has received some consideration at my hands, but I have feared this result: The test is such that the introduction of a very small quantity of nitrate would cause all the goods to be classed as nitrates, as soon as the manufacturers found what was being done.

Mr. BARTLETT. All that our State law requires is the total nitrogen. Of course we are not obliged to make the other determinations, but for a few years we made both the determination of nitric nitrogen and ammonia, and found we could not afford to do it. In adopting this plan we took 1 gram of the fertilizer and leached on the filter paper, in the same manner as for determining soluble phosphoric acid, and then determined the nitrogen in the rest. In this way we obtained the soluble nitrogen and that determination, in combination with the test for nitric nitrogen, would be valuable. The soluble nitrogen is to be stated instead of nitrogen as nitrate or ammonia.

Mr. VAN SLYKE. For several years we have been publishing water soluble nitrogen. That includes, of course, something more than nitrates, but it has satisfied the farmers and we have heard no criticism from the manufacturer.

Mr. WINTON. We have with us on this occasion a representative of the nitrate industry. A few years ago a gentleman attended the meeting who represented the ammonium sulphate industry. In our State we have always determined total nitrogen and nitrogen in the form of ammonium salts. Would it not be well to do that in an official way? In other words, would it not be well to reconsider that motion which was proposed to include ammonium salts? Not that I believe ammonium salts are equal in value to nitrate of soda, but it simply comes in the composition of a fertilizer.

Mr. DAVIDSON. I think we should determine the availability. There are a great many States that do not have a valuation for the different forms of nitrogen. There is no doubt that this is a serious defect in the laws of those States. The States ought to adopt a uniform system of stating the content of nitrogen of the various fertilizers that are put upon the market. I think we should have a law requiring, first, that all three forms should be determined, and, second, that when organic matter is stated availability also should be given. Let us add to that motion that the different forms of nitrogen be valued according to their availability, and that nitrate of soda, cotton-seed meal, dried blood, etc., be given their true valuation.

Mr. HUSTON. We have a blank for application for fertilizer analysis, on the original manufacturer's sample, and in order to relieve us a little our work was definitely stated to be on fertilizers containing nitrate. I find a large number make affidavit that the fertilizers contain nitrate when there is not a trace of nitrate in them. They have a habit of making a claim for anything which is suggested to them.

Mr. KILGORE. I would like to mention one matter, which was suggested in my report this morning, in regard to a uniform statement of fertilizer analyses and the branding of bags, etc. We had this matter up before the Association of Commissioners of Agriculture of the Southern States and they adopted part of the report, the same one that this Association has also adopted, in regard to the statement of analysis. Further, it is their opinion, on the recommendation of the chemists, that the manufacturers should make a claim of the materials out of which a fertilizer is made. A great many manufacturers objected to this, and in fact the commissioners were very doubtful as to whether such a demand should be made upon them. I believe the fertilizer committee of this Association might take up this matter, as well as that of the determination of the forms of nitrogen, and report on both subjects.

Mr. SHIVER. In order to dispose of this matter I would like to make a motion. I would like to say, in regard to the referee's report, that on account of neglect on my part we failed to have any work done on the Jones method for determining the availability of nitrogen. Those who were at the California meeting will remember that I recommended that the Jones method and the Street method be further tried by the association. I would say that I have never in my limited experience obtained very satisfactory results from the Jones method, especially with cotton-seed meal. This may be due, however, to lack of experience on my part. I make the motion that the Jones and Street methods be recommended for further trial next year.

REPORT ON PHOSPHORIC ACID.

By E. G. RUNYAN, *Referee.*

In response to a letter inviting cooperation in the work on the phosphoric acid samples for the current year, twenty-two chemists expressed a willingness to take up the work. To each was sent a set of three samples, with a copy of the following instructions:

INSTRUCTIONS FOR PHOSPHORIC ACID WORK FOR THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR 1900.

Three samples have been prepared for the work on phosphoric acid for 1900, as follows:

No. 1 is ground phosphate rock.
No. 2 is American slag.
No. 3 is ground phosphate rock.

DETERMINE TOTAL PHOSPHORIC ACID IN ALL THE SAMPLES by the following methods:

(1) *Official gravimetric method* (Bull. 46, Div. of Chem., U. S. Dept. of Agri., p. 12).

(2) *Optional volumetric method* (Bull. 46, Div. of Chem., U. S. Dept. of Agri., p. 13), or the following modification:

Make solution of the phosphate according to one of the official methods (Bul. No. 46, Div. Chem., Dept. of Agric., p. 12). Take an aliquot portion containing not more than 30 mg. of P_2O_5, add NH_4OH in excess, then HNO_3 in slight excess, then an excess of the official molybdate solution. Place in a water bath at 45° to 50° C. for one-half hour with occasional stirring, filter, and wash with water until 25 cc of the filtrate fails to neutralize one drop of the standard potassium hydroxid solution. Transfer filter and contents to the beaker, add about 30 cc of water and sufficient standard alkali to dissolve the yellow precipitate, titrating the excess of alkali with standard acid, adding 1 cc of a 1 per cent solution of phenolphthalein as indicator. See Bulletin No. 46, Division of Chemistry, Department of Agriculture (p. 13), for directions for preparing the standard acid and alkali solutions.

(3) *Volumetric method*, precipitating by shaking at room temperature.

Proceed as in the last preceding method to the point where the solution is ready to be placed in the water bath. Then cool solution to room temperature, add molybdate solution at the rate of 75 cc for each decigram of P_2O_5 present, place flask containing solution in shaking apparatus and shake for thirty minutes at room temperature, filter at once, wash and titrate as in preceding method.

DETERMINE IRON AND ALUMINUM in the three samples, by the following methods:

(1) *Modified acetate method.*—Dissolve two grams of sample in 30 cc HCl and 10 cc HNO_3 (all iron must be in the ferric condition), cool, make up to 250 cc, filter. Take 50 cc, dilute to 400 cc, nearly neutralize with NH_4HO, using a few drops of mythyl orange solution as indicator, heat to 70° C., and add 25 cc of a 25 per cent solution of ammonium acetate. Filter and wash with a hot 5 per cent solution of ammonium nitrate. Ignite and weigh the iron aluminum phosphate. Fuse the ignited precipitate with sodium carbonate, dissolve in dilute HNO_3 and determine the phosphoric acid in the usual way. Determine the iron in 50 cc of the original solution by titrating with permanganate (Margerite's method, Fresenius Quant. Anal., p. 267, ed. 1881).

(2) DETERMINE ALUMINA by precipitating with phenylhydrazine, as follows: Make solution of the phosphate in the usual way. Take an aliquot portion, dilute to 250 cc, heat to near boiling, and add ammonia slowly, as long as the precipitate formed redissolves readily. A neutral saturated solution of ammonium bisulphite is then added, drop by drop, with stirring, to the hot solution, until it becomes colorless, showing the complete reduction of the iron. Then add phenylhydrazine, a little at a time, until the precipitation is complete. The precipitate of aluminum hydroxid and aluminum phosphate is collected in a filter, washed with warm water containing a small amount of phenylhydrazine bisulphite, until the wash water is free from chlorids or from iron, as indicated by $(NH.)_2S$. Dry the filter and precipitate, ignite, heat to bright red, cool and weigh in covered crucible, as the precipitate is very hygroscopic. Fuse the mass with sodium carbonate, dissolve in hot dilute nitric acid and determine the P_2O_5 in the usual way.

The weight of the total precipitate less the weight of P_2O_5 found gives the weight of alumina. The iron is determined in another portion of the original solution by titrating with potassium permanganate.

To prepare the saturated solution of ammonium bisulphite to any given volume of strong ammonia, add an equal volume of water and pass sulphur dioxid into the cooled solution until the solution becomes yellow.

The wash solution is prepared as follows: To a few cubic centimeters of phenylhydrazine in a beaker a saturated water solution of sulphur dioxid is added gradually until the precipitate of phenylhydrazine sulphite, which at first separates out in crystals, is redissolved to a yellow solution. If after a few minutes an odor of sulphur dioxid is perceptible, a few drops of phenylhydrazine are added to neutralize this excess of sulphurous acid. Use 10 cc of this concentrated solution to 100 cc of water for washing the alumina precipitate.

When the method employed differs from directions given, please note the fact. Results on other samples will be gladly received and full credit given in the report.

Please report results at least one month before the date of the association meeting.

Results on the determination of total phosphoric acid in the samples sent out have been received from 19 analysts, representing 11 laboratories. These results are given in Table I. Where more than one result by the same method was reported by an analyst, the individual results are given in column "*a*" and the average of such results in column "*b*." When but one result was reported it is given in column "*b*."

TABLE I.—*Per cent total P_2O_5 found in referee's samples.*

Analyst.	Sample No. 1.						Sample No. 2.						Sample No. 3.					
	Official gravimetric method.		Optional volumetric method.		Volumetric by shaking at room temperature.		Official gravimetric method.		Optional volumetric method.		Volumetric by shaking at room temperature.		Official gravimetric method.		Optional volumetric method.		Volumetric by shaking at room temperature.	
	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)
J. W. Ames, Ohio Agricultural Experiment Station		14.33		14.51		14.30		18.75		17.62		17.81		27.02		27.19		26.69
A. W. Blair, Florida Agricultural College	13.67	13.69	13.65	[1]13.68			17.41	17.48	17.30	[1]17.27			26.35	26.35	26.55	[1]26.59		
	13.70		13.70				17.44		17.12				26.29		26.65			
	13.70		13.60				17.61		17.35				26.43		26.55			
			13.75				17.46		17.27				26.34		26.60			
									17.30									
									17.27									
Charles P. Beistle, Pennsylvania Agricultural Experiment Station	13.50	13.51	13.62	13.57	13.55	13.36	16.94	16.94	16.87	16.86	16.65	16.73	26.40	26.46	26.50	26.48	26.45	26.48
	13.52		13.52		13.32		16.92		16.85		16.80		26.52		26.45		26.60	
					13.20		16.97										26.40	
M. L. Bloomberg, Richmond, Va		13.46		13.58				16.84		16.86				26.10		26.17		
F. B. Carpenter, Richmond, Va				13.51						16.86						26.16		
E. M. Chace, U. S. Department of Agriculture		13.95	13.60	[1]13.73	13.63	13.61		17.15	17.40	[1]17.45	17.05	17.18		26.23	26.50	[1]26.55	26.08	27.07
			13.65		13.70				17.50		17.23				26.55		26.05	
			13.95		13.50				17.45		17.25				26.60			
E. B. Ferris, Mississippi Agricultural and Mechanical College	13.71	13.69	13.60	[1]13.66	13.45	13.48	17.39	17.47	17.30	[1]17.39	17.20	17.23	26.46	26.47	26.50	[1]26.50	26.30	26.30
	13.66		13.65		13.45		17.53		17.28		17.20		26.43		26.50		26.30	
	13.71		13.63		13.50		17.53		17.35		17.25		26.51				26.30	
			13.70		13.50		17.42		17.40		17.25						26.30	
			13.68						17.50									
			13.72						17.50									

[1] Precipitated by maintaining in bath at 45° to 50° for thirty minutes.

TABLE I.—*Per cent total P_2O_5 found in referee's samples*—Continued.

Analyst.	Sample No. 1.						Sample No. 2.						Sample No. 3.					
	Official gravimetric method.		Optional volumetric method.		Volumetric by shaking at room temperature.		Official gravimetric method.		Optional volumetric method.		Volumetric by shaking at room temperature.		Official gravimetric method.		Optional volumetric method.		Volumetric by shaking at room temperature.	
	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)
C. Glaser, Baltimore, Md.	......	13.78	......	[1]13.59	......	13.26	......	[4]14.52	......	[5]13.99	......	[4]14.09	......	26.12	......	26.82	......	25.90
	......	......	......	......	......	......	......	......	......	......	......	......	......	......	......	[6]26.12	......	......
O. W. Knight, Maine Agricultural Experiment Station	14.23	14.22	......	14.21	......	14.16	17.99	18.06	......	18.03	17.82	17.82	27.08	27.12	......	27.19	27.04	27.02
	14.21		......	......	......	......	18.04		......	......	17.82		27.16		......	......	26.99	
	......	......	......	......	......	......	18.14		......	......	......	......	......	......	......	......	......	......
F. C. Lamb, Raleigh, N. C.	13.47	13.43	......	......	......	......	17.22	17.25	......	......	......	......	25.96	25.95	......	......	......	......
	13.46		......	......	......	......	17.19		......	......	......	......	25.98		......	......	......	......
	13.39		......	......	......	......	17.33		......	......	......	......	25.91		......	......	......	......
	13.38		......	......	......	......	......	......	......	......	......	......	......	......	......	......	......	......
J. R. Laughlin, Maryland Agricultural College	13.56	13.64	13.40	[2]13.46	......	......	17.31	17.29	17.05	[2]17.09	......	......	25.80	25.90	25.75	[2]25.82	......	......
	13.67		13.48		......	......	17.31		17.10		......	......	25.93		25.90		......	......
	13.69		13.50		......	......	17.25		17.12		......	......	25.97		25.80		......	......
C. C. McDonnell, Clemson Agricultural College, South Carolina	......	13.86	......	[1]13.50	......	13.31	......	17.41	......	[1]17.13	......	17.13	......	26.24	......	[1]26.00	......	26.04
H. K. Miller, Florida Agricultural College	13.75	13.92	13.85	[1]13.87	13.75	13.70	17.62	17.47	17.35	[1]17.41	17.30	17.35	26.52	26.53	26.70	[1]26.66	26.80	26.64
	13.84		13.80		13.65		17.44		17.40		17.40		26.62		26.70		26.65	
	13.89		13.85		......	......	17.38		17.35		......	......	26.31		26.85		26.65	
	14.05		13.75		......	......	17.42		17.30		......	......	26.52		26.80		26.45	
	14.00		13.80		......	......	......	......	17.35		......	......	26.38		26.80		......	......
	13.98		13.80		......	......	......	......	17.30		......	......	26.82		26.75		......	......
	......	......	14.05		......	......	......	......	17.45		......	......	......	......	26.60		......	......
	......	......	14.05		......	......	......	......	17.40		......	......	......	......	26.60		......	......
	......	......	14.10		......	......	......	......	17.60		......	......	......	......	26.60		......	......
	......	......	13.72		......	......	......	......	17.50		......	......	......	......	26.60		......	......
	......	......	13.80		......	......	......	......	17.35		......	......	......	......	26.55		......	......
	......	......	13.90		......	......	......	......	17.30		......	......	......	......	26.70		......	......
	......	......	13.85		......	......	......	......	17.55		......	......	......	......	26.55		......	......
	......	......	13.90		......	......	......	......	17.35		......	......	......	......	26.60		......	......
	......	......	13.90		......	......	......	......	......	......	......	......	......	......	26.55		......	......
	......	......	......	......	......	......	......	......	......	......	......	......	......	......	26.65		......	......

E. L. Parker, Richmond, Va	...	13.60	...	13.60	...	...	...	16.91	...	16.82	...	...	...	26.15	...	26.15	...	...
B. F. Robertson, Clemson Agricultural College, South Carolina	...	13.90	...	[1]13.56	...	13.68	...	17.46	...	[1]17.18	...	17.18	...	26.21	...	[1]26.19	...	26.04
E. G. Runyan, U. S. Department of Agriculture	13.59	13.61	13.60	[1]13.60	13.70	13.69	17.23	17.26	17.40	[1]17.42	17.30	17.36	26.03	26.14	26.70	[1]26.62	26.70	26.67
	13.68		13.60		13.90		17.29		17.30		17.40		26.22		26.60		26.80	
	13.65		13.60		13.80		17.29		17.50		17.35		26.09		26.60		26.75	
	13.65		...	...	13.80		17.23		17.40		17.30		26.20		26.75		26.60	
	13.63		...	...	13.50		...	...	17.30		17.40		...	...	26.50		26.50	
	13.53		...	...	13.50		...	...	17.50		17.40		...	...	26.55		...	...
	13.53		...	...	13.60		...	...	17.55		...	...	...	...	...	...	...	...
	...	...	...	...	13.70		...	...	...	...	...	...	...	...	...	...	...	...
M. N. Straughn, Maryland Agricultural College	13.48	13.54	13.45	13.53	...	...	17.27	17.32	17.25	17.18	...	...	25.93	25.99	25.80	25.88	...	...
	13.51		13.55		...	...	17.32		17.10		...	...	26.05		25.95		...	...
	13.64		13.60		...	...	17.37		...	...	...	...	...	...	...	...	...	...
W. S. Welch, Mississippi Agricultural and Mechanical College	13.80	13.77	13.73	13.73	13.50	13.51	17.56	[3]17.55	17.35	17.34	17.15	17.20	26.51	26.47	26.50	26.53	26.25	26.25
	13.74		13.73		13.52		17.59		17.35		17.23		26.43		26.50		26.25	
	...	...	13.75		...	...	17.49		17.33		17.23		...	...	26.55		...	...
	...	...	13.70		...	...	17.55		17.35		...	...	...	...	26.50		...	...
	...	...	13.75		...	...	17.56		17.30		...	...	...	...	26.60		...	...
	...	...	13.73		...	...	...	...	17.33		...	...	...	...	26.50		...	...
	...	...	...	...	...	...	...	...	17.33		...	...	...	...	...	...	...	...
	...	...	...	...	...	...	...	...	17.35		...	...	...	...	...	...	...	...
C. B. Williams, Raleigh, N. C	13.55	13.51	...	...	13.45	13.49	17.21	17.26	...	...	16.88	16.92	26.01	26.06	...	...	25.90	25.86
	13.43		...	...	13.50		17.23		...	...	16.93		26.10		...	...	25.[illegible]0	
	13.55		...	...	13.50		17.27		...	...	16.90		26.11		...	...	25.85	
	13.51		...	...	13.51		17.27		...	...	16.95		26.02		...	...	25.88	
	...	...	...	...	...	...	17.33		...	...	...	...	...	...	...	...	...	...
Averages	...	13.75	...	13.70	...	13.63	...	17.40	...	17.24	...	17.26	...	26.31	...	26.44	...	26.41
Number arranged	...	18	...	17	...	12	...	17	...	16	...	11	...	18	...	17	...	12

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[1] Precipitated by maintaining in bath at 45° to 50° for thirty minutes.
[2] Precipitated by maintaining in bath at 65° for thirty minutes.
[3] Contaminated with iron.
[4] Not included in averages.
[5] Precipitated by maintaining in bath at 45° to 50° for thirty minutes; not included in averages.
[6] Washed with water at 50°; not included in averages.

COMMENTS BY ANALYSTS.

C. P. Beistle.—The two volumetric methods were slower than the official gravimetric method, and also gave less uniform results. A great deal of time was required in washing the yellow precipitate with 10 per cent nitric and 3 per cent ammonium nitrate, and water successively. If the precipitate was washed with water alone the results were invariably high, indicating the presence of molybdic acid in the precipitate. In precipitating the phosphoric acid at room temperature with shaking, some of the precipitate adhered to the sides of the flask, and could not be removed.

C. Glaser.—Concerning the titration of the yellow precipitate, I wish to say that the method is very good, but I can not rely upon getting always the same results. I do not succeed in washing out completely with cold water, but tepid water (50°) does very well.

J. R. Laughlin.—In the modified volumetric method I had trouble with the yellow precipitate running through filter. This was remedied by precipitating at higher temperature, or leaving in the bath for a longer time than given in the referee's directions. I precipitated for 30 minutes at 65°.

C. B. Williams.—The solution of the three samples of the referee's phosphoric acid was effected by first boiling 2 grams each in 200 cc flasks with 30 cc of hydrochloric acid (1.20 specific gravity) to a concentration of 10 or 15 cc, and then adding, after slightly cooling, 30 cc nitric acid (1.42 specific gravity), and again boiling until the cessation of the evolution of red fumes, which required about 10 or 20 minutes. This is the method used in this laboratory for effecting the solution of phosphoric acid in slags and fertilizers containing iron and alumina.

The volumetric results were obtained by precipitating in the cold in Erlenmeyer flasks with molybdic solution, and then placing the flasks in a revolving shaking machine for 30 minutes, 45 to 55 revolutions per minute being maintained. This method of precipitating gives a coarse precipitate that can be filtered and washed easily and quickly. It will be observed that the gravimetric results on sample No. 2 are perceptibly higher than those volumetrically determined. This is probably due to the presence of iron in the magnesium pyrophosphate, as a qualitative test for iron revealed its presence in considerable quantities in the "white precipitate."

OBSERVATIONS BY REFEREE.

By a comparison of the data reported it was found that all results by Mr. Glaser on No. 2, the sample of slag, were about 3 per cent lower than those reported by other analysts. A correspondence with Mr. Glaser brought out the fact that the solution of this sample in aqua regia was evaporated to dryness, and the residue taken up with hot dilute hydrochloric acid to separate the silica. This subsequent treatment with hydrochloric acid ought to have resulted in a solution of all the phosphoric acid, but it appears that in this case it failed to do so. These results are given in the table with this explanation, and will serve to emphasize a fact that has been observed before in the work of this association; that is, a slight variation in the method may cause a wide difference in the result.

By a reference to the table it will be seen that the results reported by two of the analysts are uniformly high in comparison with the other results received. With these exceptions the percentages by the several methods agree fairly well, and may be considered quite satisfactory. The gravimetric results in the sample of slag are slightly higher than those by the volumetric method, due probably to the presence of iron in the white precipitate. For this reason it is quite probable that the volumetric results on such material more nearly express the true percentage of phosphoric acid present.

One-half the analysts reporting on the optional volumetric method state that the precipitation was made in a bath at 45° to 50° for 30 minutes, as indicated in the table by a reference to footnote "1". This is the method of precipitation usually employed in the laboratory of the Division of Chemistry, as we have found that the yellow precipitate formed under these conditions filters rapidly and there is less danger of its contamination with molybdic acid.

Two years ago Mr. Kilgore, then referee on phosphoric acid, presented to this association some results obtained by precipitating the phosphoric acid at room temperature by shaking. One advantage claimed for this method was that there was less liability of the precipitate being contaminated with molybdic acid. The results reported at that time were so promising for this method that I requested a trial of it by the chemists taking part in the work this year. An inspection of Table I shows that while the results by shaking in the cold are very slightly lower, still they agree well with results by the other two methods. One analyst reports that in precipitating by shaking in the cold, a portion of the yellow precipitate adhered to the flask and could not be removed. This objection can be overcome by dissolving the adhering precipitate in a portion of the standard alkali, and then rinsing the contents of the flask into the beaker in which the titration is made, or by transferring filter and contents to the flask and making the titration in the flask.

Some trials were made by your referee to determine the time required to precipitate all the phosphoric acid by shaking in the cold. On several different days determinations were made in quadruplicate, one set of four shaken for 30 minutes and the other set shaken for 1 hour. All results agreed closely. The average results by the two periods of shaking differed by less than 0.05 of 1 per cent, showing that all the phosphoric acid was precipitated at the end of 30 minutes. Judging from my own experience and from the data reported by others, I believe this method capable of yielding good results in the hands of any careful analyst.

Some results, obtained by modifications of the official method, were reported by Mr. Welch on the referee's samples, on an acid phosphate practically free from iron, and on chemically pure sodium phosphate. These figures are given in Table II.

TABLE II.—*Results on phosphoric acid by modifications of official methods. By W. S. Welch.*

Methods.	Referee's samples.			Acid phosphate.	C.p. sodium phosphate containing 19.83 per cent P_2O_5.
	No. 1.	No. 2.	No. 3.		
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Official gravimetric	13.75	17.75[1]	26.47	17.88 17.91	
Official gravimetric after precipitating the "yellow" precipitate by shaking 30 minutes at room temperature				17.86 17.91	
Official gravimetric, adding 5 cc ammonium citrate solution before adding magnesia mixture	13.56 13.64	17.36 17.44	26.28	17.76 17.72	
Official gravimetric, adding 10 cc ammonium citrate solution before adding magnesia mixture				17.73 17.71	
Official gravimetric, adding 2 grams citric acid to 0.25 gram sample before adding magnesia mixture		17.32 17.33			
Volumetric, shaking 30 minutes at room temperature	13.51	17.20	26.25	17.70 17.80 17.75 17.83 17.80 17.80	19.85
Optional volumetric	13.73	17.34	26.53		
Official gravimetric, "white" precipitate dissolved in HCl, reprecipitated with NH_4OH				17.64 17.67	

[1] Contaminated with iron.

IRON AND ALUMINA IN PHOSPHATES.

Results on iron and alumina in the phosphoric-acid samples sent out were received from seven analysts representing as many laboratories. These results are brought together in Table III.

TABLE III.—*Results on iron and alumina in referee's samples.*

Analysts.	Sample No. 1.						Sample No. 1.						Sample No. 3.					
	Per cent Fe_2O_3 by permanganate.		Per cent Al_2O_3.				Per cent Fe_2O_3 by permanganate.		Per cent Al_2O_3.				Per cent Fe_2O_3 by permanganate.		Per cent Al_2O_3.			
			Acetate method.		Phenylhydrazine.				Acetate method.		Phenylhydrazine.				Acetate method.		Phenylhydrazine.	
	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)
J. W. Ames, Ohio Agricultural Experiment Station		4.78		7.00				18.05		0.21				5.65		7.30		
C. P. Beistle, Pennsylvania Agricultural Experiment Station	4.52 4.45	4.49	8.22 8.27	8.25														
A. W. Blair, Florida Agricultural College		4.78	7.50 7.40 7.38 7.48	7.44				17.83	2.41 [5]6.71 1.31 1.07	1.60				4.24	5.16 8.10 7.93 8.00	7.30		
E. B. Ferris, Mississippi Agricultural and Mechanical College	3.37 3.43	3.40	8.35 8.32 8.35	[1]8.34									4.00 4.00 4.00	4.00	5.22 5.08 4.81	[1]5.04		
C. Glaser, Baltimore, Md.		6.28		6.52		8.45		21.22				0.67		5.98		6.97		5.95
E. G. Runyan, United States Department of Agriculture	4.33 4.28 4.33 4.43	4.34	7.86 8.01 8.34	8.07	8.98 8.40 8.85	8.74	18.23 18.18	18.21	1.84 1.92 1.97	1.91	1.25 1.58 1.75 1.75	1.58	5.09 4.99 5.04	5.04	8.44 8.24 8.64	8.44	5.88 6.23 5.75	5.95
S. H. Sheib, Richmond, Va		5.00		[2]7.10 [3]6.47 [4]6.90				18.12		[2]1.22 [3]0.06 [4]0.01				5.75		[2]6.05 [3]4.55 [4]4.16		
Average		4.72		7.46		8.59		18.69		1.24		1.12		5.11		7.50		5.95
Difference between highest and average		1.56		.79		.15		2.53		.67		.46		.87		.94		
Difference between lowest and average		1.32		.94		.14		.86		1.03		.45		1.11		.53		
Number averaged		7		5		2		5		3		2		6		4		2

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[1] Precipitated twice, with addition of a little sodium phosphate the second time; not included in averages.

[2] Contained lime; not included in averages.

[3] Precipitated twice; not included in averages.

[4] By acetate method as used in laboratory of Virginia-Carolina Chemical Co.; not included in averages.

[5] Not included in averages.

COMMENTS BY ANALYSTS.

F. B. Carpenter (*S. H. Sheib*).—The iron and alumina determinations were made by the modified acetate method, as described in the referee's letter of instructions, and also by the modified acetate method used in this laboratory, which is as follows:

Weigh two grams of sample in a 200 cc flask, add 35 cc strong hydrochloric acid and boil thirty minutes. Dilute, cool, make to mark, mix, filter through dry filter. Take 50 cc for analysis, add a few drops nitric acid, boil, add ammonia to alkalinity and a few drops of methyl orange, then acidify slightly with hydrochloric acid. Heat to 70° and add 15 to 20 cc of a strong hot solution of ammonium acetate made distinctly acid with acetic acid. Filter at once on folded filter, wash three or four times with a 5 per cent solution of ammonium nitrate and once with hot water. Dissolve the precipitate with dilute warm hydrochloric acid and reprecipitate the iron and alumina as above. Filter, wash six times with 5 per cent ammonium nitrate solution, dry, ignite, and weigh as iron and aluminum phosphate. When but one precipitation was made in the work on the referee's samples, the iron and aluminum phosphates contained heavy traces of lime, while two precipitations reduced the lime present to a slight trace.

C. Glaser.—The determination of the combined iron and aluminum oxids in sample No. 2, by precipitation of the iron and aluminum phosphate with acetate, proved an utter failure on account of lack of phosphoric acid. The phenylhydrazine process is excellent, but I can not account for the difference in results obtained, No. 1 being too high and No. 3 being too low.

OBSERVATIONS BY REFEREE.

The results on iron and alumina are rather discordant, although they show slightly closer agreement than did the results obtained the last time this subject was before this association.

The phenylhydrazine method is one proposed by Hess and Campbell, and your referee requested a trial of it in the hope that it might yield concordant results in the hands of the members of this association. As only one analyst outside of the Department of Agriculture reported results by this method, it would be manifestly unwise to base any conclusion on such meager data.

The other method given was the well-known acetate method with a slight modification. Two fertile sources of error by the acetate method, as usually employed, are the solvent action of the wash water on the precipitate of iron and aluminum phosphate, and the contamination of the precipitate with lime. The modification proposed sought to reduce the solvent action to a minimum by washing with a 5 per cent solution of ammonium nitrate and to avoid the error due to the presence of lime by taking not more than 0.4 gm of the sample and diluting to 400 cc before precipitation.

In some preliminary trials of this method in the laboratory of the Division of Chemistry, on solutions of chemically pure salts, it was observed that no lime was precipitated by ammonium acetate in a faintly acid solution when the dilution was carried to the point mentioned above.

Some results by the two proposed methods on a solution of chemically pure aluminum sulphate and a mixture of chemically pure salts containing known quantities of alumina, iron, lime, and phosphoric acid are given in Table IV.

Table IV.—*Results on alumina in mixtures of chemically pure salts, by E. G. Runyan.*

Methods.	Mixture chemically pure salts containing Fe_2O_3, P_2O_5, CaO, Al_2O_3.		Chemically pure aluminum sulphate.	
	Per cent Al_2O_3.		*Per cent Al_2O_3.*	
Theory		4.85		19.38
Phenylhydrazine method	5.14	5.17	18.84	19.16
	5.18		19.04	
	5.18		19.60	
Modified acetate method	4.70	4.67	19.63	19.50
	4.60		19.25	
	4.72		19.63	
	4.65		....	

The results on the chemically pure aluminum sulphate do not agree as closely as one could wish, while the average of the results on the mixture of pure salts by the phenylhydrazine method are 0.32 of 1 per cent too high, and by the acetate method 0.18 of 1 per cent too low.

Although the results reported on iron by the permanganate method do not agree as well as they should in the hands of experienced analysts, still I believe this method is the best one for the determination of iron in phosphates. As the alumina by the acetate method is determined by difference, it follows that an error in the determination of iron or phosphoric acid would cause a corresponding error in the percentage of alumina found. Some of the apparent discrepances in the percentages of alumina as reported by the acetate method may be due to errors in the determination of iron. While the results given do not warrant the drawing of any definite conclusions, still I believe that some form of the acetate method will prove to be the best method for alumina.

The subject of the determination of iron and alumina in phosphates is one that, in my opinion, may well receive some attention from the members of this association during the coming year.

Mr. Wheeler. I should like to present a paper for the purpose of calling the attention of the association to some work which has been done on phosphoric acid.

Mr. Wheeler presented his paper as follows:

INCREASED ACCURACY IN PHOSPHORIC ACID DETERMINATION.

By H. J. Wheeler.

Berzelius[1] recognized the existence of an ammonium magnesium phosphate of the ortho type, richer in ammonia than the normal one. Subsequently Neubauer,[2] apparently unaware of Berzelius's observation, directed attention to such a salt, and, in recognition of its existence and the consequent error liable to arise in the determination of phosphoric acid, proposed the use of certain factors to correct the errors in analysis.

Gooch and Austin[3] have shown that if the ammonium magnesium phosphate is precipitated in the absence of an excess of ammonium hydroxid and ammonium salts and under definite conditions the normal salt is obtained. Their procedure obviates the necessity of applying factors for correcting results. It does away, also, with the necessity of adding magnesia mixture drop by drop in an attempt to bring about a compensation of errors.

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[1] Jahresb., 3 Jahrgang (1824) Uebersetzt von C. G. Gmelin, p. 92. Cited from Gooch and Austin.

[2] Zeit. f. Angw. Chemie (1896) pp. 435–440.

[3] Am. Jour. of Science 7, pp. 187–198.

There seem to be three excellent reasons why this association should give special consideration to the work of Gooch and Austin and why certain modifications of the present official method of determining phosphoric acid are advisable.

(1) By a modification of the method in accordance with their procedure the laboratory may be freed from the strong odor of ammonia.

(2) The laboratory expenses may be materially reduced by lessening the amounts of ammonium chlorid and ammonium hydroxid required.

(3) By the proposed modification close approximations to theoretical results are obtained, since the final precipitation is always conducted under essentially identical conditions.

As determinations are now conducted by the official method the large quantities of ammonium hydroxid and of ammonium salts, together with their varying relation to the magnesium salt present, render it impossible either to obtain the ideal salt or to secure a condition under which the deficiency of magnesia in the residue is compensated for by the magnesium oxid thrown down. According to the method of the association, one is directed to dissolve the yellow precipitate "on the filter with ammonia and hot water, and wash into a beaker to a bulk of not more than 100 cc." Hydrochloric acid is then added until the solution is nearly neutral, before precipitating with magnesia mixture. Under such conditions large amounts of ammonium salts are not only present in all cases, but the quantity is necessarily variable. The existing conditions are further aggravated by the large quantities of ammonium hydroxid and ammonium chlorid in the official magnesia mixture.[1] After the precipitation with magnesia mixture is completed, the method of the association calls for the addition of 30 cc of ammonium hydroxid of 0.96 specific gravity. Since the strength of the ammoniacal solution and the quantity of ammonium salts at the time of precipitation is seldom twice alike, it must be obvious that if a nearly correct result is obtained finally, it must be due to chance and to a balancing of errors.

In addition to the points which have been enumerated, Gooch and Austin have shown that the double magnesium and ammonium phosphates are extremely insoluble, even in a faintly ammoniacal solution, and that such a solution may be used for washing the precipitate in the place of the disagreeably strong one provided for in the official method. The following is a modification of the official method whereby a precipitate of ideal composition may be obtained:

After dissolving the yellow precipitate in the usual manner the solution is allowed to cool (with care the precipitation may be made immediately), and the phosphoric acid precipitated with magnesia mixture made as directed by Blair. If it seems advantageous, the solution may be nearly neutralized with hydrochloric acid before precipitating. The magnesia mixture may be added at once instead of drop by drop. As soon as the precipitate subsides, the supernatent liquid is decanted upon the same asbestus filter upon which the final precipitate is to be collected. Most of the liquid may thus be poured off without the transference of more than a minimum portion of the first precipitate to the filter. The precipitate is dissolved in the least possible amount of hydrochloric acid with the addition of 75 cc to 100 cc of distilled water. Ammonium hydroxid is added until the solution reacts distinctly alkaline. The precipitate, even where but small quantities of phosphoric acid are present, is formed immediately. It subsides quickly and can be filtered in a few moments, or as soon as the solution is cool. The wash water should be rendered faintly alkaline with ammonium hydroxid.

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[1] Two liters contain 280 grams of ammonium chlorid and 700 cc of ammonium hydroxid (0.96 specific gravity). Blair's mixture, on the other hand, contains in the same volume but 58 grams of ammonium chlorid and only sufficient ammonium hydroxid to give it a distinct odor of ammonia.

The following are a few data obtained by Miss Austin at the Rhode Island station in the analysis of ordinary commercial fertilizers by the official method and by the modification just described:

Comparison of methods in determining phosphoric acid.

Fertilizer No.		Official method.	Modified method (Blair's mixture).	Modified method: Precipitation from the warm ammoniacal solution (Blair's mixture).
		$Mg_2P_2O_7$ *grams.*	$Mg_2P_2O_7$ *grams.*	$Mg_2P_2O_7$ *grams.*
284	Solution I	.0742	.0720	....
		.0728	.0712	....
	Solution II	.0740	.0718	....
		.0724	.0720	....
286	Solution I	.0718	.0734	.0730
		.0714	.0738	.0732
	Solution II	.0720	.0734	.0732
		.0716	.0734	.0734
288	Solution I	.0788	.0788	.0788
		.0794	.0790	.0786
	Solution II	.0785	.0766	.0770
		.0786	.0762	.0772
318	Solution I	.0146	.0148	....
		....	.0146	....
	Solution II	.0144	.0146	....
		....	.0146	....

It will be seen from the foregoing that in some instances the results by the modified method were higher and in others lower than by the official method, but a better average agreement between parallel tests was obtained by the modified than by the official method.

When one considers that by the proposed modifications the magnesia mixture may be added at once instead of drop by drop, also that a great saving in ammonium chlorid, ammonium hydroxid, and hydrochloric acid may be effected, and that the disagreeably strong ammoniacal wash water may be replaced by a very weak one, there are abundant reasons, in addition to the attainment of greater accuracy, why the question of changing the official method is worthy of the serious consideration of this association.

Mr. Macfarlane. An apology is due this association for introducing the vexed question of available phosphoric acid. My paper is on that perplexing subject. I may say that we have in Canada, for the past twelve years at least, been loyal to Bulletin 46, and in fact every one of our analysts throughout the Dominion is furnished with a copy of the work in question, and its use is recommended and in fact urged. We have had no occasion during the period mentioned to suggest any changes, but it has happened that our peace paradise has now been disturbed. We have had in Canada very large importations of basic slag, or Thomas phosphate, which I understand is very seldom imported into the United States, and the consequence is we have gotten into trouble. We are obliged to do something to give satisfaction both to consumers and manufacturers, not only of basic slag, but of other acidulated phosphates that may be sold in the Dominion.

Under our law we collect samples and they are submitted to analysis. Our district analysts applied the processes described in your methods of analysis to the basic slags, the consequence being that the amount of available phosphoric acid, which the law requires in phosphates sold in Canada, was perhaps underestimated. It did not come up to the 8 per cent of available phosphoric acid which, by law, the fertilizers in Canada should contain. Instead of having the supposed 8 per cent, it sometimes did not amount to more than 4 or 5 per cent, and the consequence was that the district analysts were obliged to characterize the basic slags which were being sold in the Dominion as adulterated, according to the act. Of course, this was not at all agreeable to the importers or venders of the basic slag, and we have heard a great deal of discussion, which culminated this last session of Parliament, in an appeal, as it were, to the agricultural committee of the House of Congress. What actually happened before that agricultural committee is detailed in our last bulletin, of which I have some copies here, and which I think a good many members of the association received directly. I shall therefore not refer again to what took place before that committee, but I will try to describe the action which I took subsequently. In order that I may not omit any of the important points, allow me to read from this memorandum:

MEMORANDUM FOR THE MEETING OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS AT WASHINGTON, D. C., ON THE 16TH OF NOVEMBER, 1900.

On pages 56 and 57 of Bulletin No. 70, issued by the Laboratory of the Inland Revenue Department at Ottawa, there will be found a statement regarding the present position of the question which has arisen in Canada as to the proper method of estimating the "available" phosphoric acid in basic slag, or Thomas phosphate powder. The importers of this article, represented by James Domville, esq., M. P. for Kings County, New Brunswick, did not accept the amendment to the fertilizers act proposed by the minister of inland revenue and approved by the agricultural committee of the House of Commons, and consequently the public analysts of Canada will find themselves face to face next year with the same conditions which now obtain, and be obliged to characterize nearly all samples of basic slag as "adulterated under the act."

Foreseeing the inconveniences of such a result, I took advantage of my stay in London, England, during the months of June and July, this year, to make an application to the Society of Public Analysts there for information as to whether it would be likely to take any action in the direction of establishing a uniform method for the determination of the available phosphoric acid in fertilizers. This application was considered at a meeting of the council of the society, and the following gentlemen appointed a committee to consider the subject: Dr. Bernard Dyer, Dr. Voelcker, Messrs. John Hughes, and Alfred Sinesham. Owing to the lateness of the season, it was found impossible to arrange for a meeting of this committee in July, and the matter had therefore to stand until after the recess, with what result since I am unable to say.

While in London I thought it wise also to call on the firm of H. & E. Albert, chemical works, one of the largest shippers of basic slag to Canada, in order thoroughly to explain the position of our branch and why their wish could not be complied with, namely, that the Wagner method of determining the available phosphoric acid should be adopted in our laboratory. My arguments were mainly founded on the position taken under paragraph 5 on page 6 of Bulletin No. 70, which is as follows:

> To apply a 2 per cent citric-acid solution in the manner described by Wagner to basic slag only and not to the water-insoluble part of other fertilizers would, without doubt, occasion strong objections on the part of the fertilizer manufacturers of Canada and of the United States.

I did not content myself with defending this position, but also suggested a method by which all fertilizers could be tested in exactly the same way without doing any injustice to basic slag or Thomas phosphate powder. This consisted in boiling the water-insoluble residue with a strong solution of ammonium chlorid, so as to remove any free lime which the sample might contain, and to do this before determining the available phosphoric acid, either by neutral citrate of ammonia or by a 1 per cent solution of citric acid, as recommended by Dr. Bernard Dyer and others. The gentlemen to whom I spoke in the London office of Albert's works proposed that I should discuss the method suggested with the chemists of the firm at Biebrich on the Rhine and with Professor Wagner in Darmstadt. This I ultimately consented to do, and the London house arranged for appointments with the parties named.

My interview with Dr. Wagner took place on July 12, and resulted most satisfactorily. We discussed the fertilizer act of the Dominion, the whole circumstances connected with the analysis of basic slag, and also my suggestion for treating it with a solution of ammonium chlorid. The latter he declared to be new and well worth a minute investigation, which he thought should be undertaken after consultation with the Albert firm. The authorities of the latter I also visited at their works at Biebrich, including not only Mr. Heinrich Albert, but the manager also, and the chief chemist, Mr. König. Mr. Albert made many objections to my proposal, more especially maintaining that the ammonium chlorid solution would dissolve out phosphoric acid. Experiments by Mr. König showed this fear to be unfounded, while at the same time much lime was removed, and a large quantity of phosphoric acid dissolved out by a 1 per cent citric acid solution. It was at last agreed that the new method should be thoroughly tested, both by the Biebrich chemist and by Dr. Wagner, and the results communicated to the London house as early as possible.

In October, after my return to Canada, I caused some trials to be made on basic slag, using the process above mentioned for removing free lime, and now desire to mention some of the results to this association. Three samples were experimented on, being Nos. 1128, 1146, and 1147 of Bulletin No. 70. Their contents in phosphoric acid, as claimed by the venders, are given first in the following table, and then the results of the successive treatments to which they were subjected.

Comparison of methods in determination of phosphoric acid in basic slag.

Sample No.	1128.	1146.	1147.	16774.	19336.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Total phosphoric acid claimed	17.00	18.32	18.18	....	....
Available phosphoric acid claimed, determined by Wagner's process and 2 per cent solution	....	15.87	15.87	....	....
Total phosphoric acid found	18.00	18.30	17.60	13.11	16.06
Available phosphoric acid, found by the ordinary United States citrate treatment	6.90	8.35	6.83	6.82	5.83
Lime (stated as CaO) dissolved out by boiling 5-gram sample for sixty minutes with 20 grams ammonium chlorid in 200 cc of water, replacing the loss of the latter; "A" treatment	7.53	7.01	8.06	....	....
Lime dissolved by boiling 5-gram sample for thirty minutes with 5 grams chlorid ammonium in 100 cc water, replacing latter; "B" treatment.	6.74	5.56	7.57	....	....
Lime dissolved by boiling 5-gram sample for sixty minutes with 5 grams chlorid ammonium in 50 cc water, replacing latter; "C" treatment	8.18	5.76	8.29	6.52	6.43
Phosphoric acid remaining in residue from "A" after treating it with citrate of ammonia by the United States official method	6.33	6.91	7.16	Lost.	5.63
Consequently available phosphoric acid by the "A" treatment	11.67	11.39	10.84	....	10.43
Phosphoric acid remaining in the residue from "B," after occasional agitation for 30 minutes in the cold with 500 cc of 1 per cent citric acid solution	11.39	7.04	5.87	....	....
Consequently available phosphoric acid by the "B" treatment	6.61	11.26	12.13	....	....
Phosphoric acid remaining in the residue from "C," after energetic shaking in a horizontal mechanical agitator for 30 minutes, with 500 cc of 1 per cent citric-acid solution	4.60	3.20	3.58	3.45	2.69
Consequently available phosphoric acid by the "C" treatment	13.40	15.10	14.42	9.66	13.37
Recapitulation as regards available phosphoric acid without boiling in chlorid ammonium	6.90	8.35	6.83	6.82	5.83
By treatment "A"	11.67	11.39	10.84	Lost.	10.43
By treatment "B"	6.61	11.26	12.13	....	....
By treatment "C"	13.40	15.10	14.42	9.66	13.37
By Wagner method	....	15.87	15.87	....	....

It will be observed from the experiments just described that from 5½ to 8¼ per cent of lime were dissolved out of the samples by the ammonium-chlorid solution, and that this occasioned an average increase of nearly 4 per cent in the citrate soluble or available phosphoric acid as determined by the ordinary official citrate of ammonia method. When 200 cc of 1 per cent citric-acid solution is substituted for the citrate of ammonia (100 cc of a 20 per cent solution) and allowed to act in the cold with moderate agitation, the increase is about the same. When, however, vigorous mechanical agitation is employed the increase in the citric soluble or available amounts to 7 per cent. To me it does not appear reasonable that mechanical agitation should be used, that being so very unlike what obtains in nature.

The modification which I have to suggest in the official methods under "3. Determination of phosphoric acid (4) and (5)" on p. 13 of "Methods of analysis," Bulletin No. 46, revised edition, is as follows:

(4) Citric insoluble phosphoric acid.

(*a*) In acidulated samples: Introduce the filter containing the washed residue from the water-soluble phosphoric acid determination into a flask with 100 cc of 1 per cent citric acid solution. Stopper tightly and shake violently until the filter paper is reduced to a pulp. Add 100 cc additional of the 1 per cent citric-acid solution and digest in the cold for 30 minutes. Shake the flask every five minutes. Filter and wash thoroughly. Dry and transfer the filter and its contents to a crucible, ignite until all organic matter is destroyed, add from 10 to 15 cc of strong hydrochloric acid and digest until all phosphate is dissolved; or return the filter with contents to a digestion flask, add from 30 to 35 cc of strong nitric acid and from 5 to 10 cc of strong hydrochloric acid and boil until all phosphate is dissolved. Dilute the solution to 200 cc, mix well, filter through a dry filter, take a definite portion of the filtrate, and proceed as under total phosphoric acid.

(*b*) In nonacidulated samples: In case a determination of citric-insoluble phosphoric acid is required in nonacidulated samples, such as basic slag, ground bone, bone ash, etc., it is to be made by taking 2 grams of the phosphatic material (without previous washing with water) and introducing it into a flask with 100 cc of a 5 per cent solution of chlorid of ammonium and boiling it for 30 minutes, replacing always the evaporated water; then filtering and washing the residue and treating it exactly as above described with 1 per cent citric acid solution. In case the substance contains much animal matter (bone, fish, etc.) the residue insoluble in citric acid is to be treated by one of the processes described under total phosphoric acid. a_2, a_3, or a_4.

5. Citric-soluble phosphoric acid.

The sum of the water-soluble and the citric-insoluble subtracted from the total gives the citric-soluble phosphoric acid. The sum of the latter and the water-soluble phosphoric acid is to be regarded as "available" phosphoric acid.

I make this suggestion with all due deference only for what it is worth, and in order that this association may have a definite proposal before it in the event of its deciding to investigate the subject further. I would respectfully represent that the matter does deserve investigation at your hands, and I believe that if it is undertaken the result will be a decided step toward establishing such methods of analysis as will obtain acceptance not only in the United States but also in British countries and on the continent of Europe.

Mr. Myers. I am very glad that Mr. Macfarlane has brought up this subject and offered some basis upon which this convention may take action. Some three months ago I was asked by a gentleman of the Thomas Phosphate Propaganda in Berlin to bring before this convention the question of the analysis of Thomas phosphate imported into this country. We are all aware that the phosphoric acid in Thomas slag is in a form that gradually becomes available when applied to lands of a certain character, whereas our methods of analysis in this country fail to show the actual value of the phosphoric acid present in the compound. In other words, our methods in this country are adapted for the analysis of acid phosphates and for such phosphates as are produced in our processes of manufacture here on a large scale, but are not adapted to this by-product of another manufacturing process, although the Thomas phosphate is a product that may come into this country on a large scale soon; and in fact is already coming in to a considerable extent at two or three points. There is none at all being received along the Atlantic coast, but large quantities are used in Texas and California and the consumption is likely to increase on the Pacific coast. Those interested would like this association to take steps to improve their methods of analysis of this product. It is simply justice to them. They are not adulterating their goods, but the difficulty is in the processes of analysis. The function of the office of official chemist, as I conceive it, is not to prevent the sale of an article, but it is to prevent its adulteration. These goods, as a rule, are not adulterated, but our processes of analysis are such that they can not get the proper valuation, and they do not show their real worth. What they ask is that this association should take up this matter, investigate it, and settle upon some method of analysis which will be mutually satisfactory to the State chemists of this country and to the producers of this product. It is a reasonable request, and I am confident that the association will instruct its reporter next year, at least, to take up this question and go to the bottom of it so far as he can, and in the meantime make provision so that the chemists in the States of Texas and California and in Canada may have some means of properly controlling the inspection of these goods.

The PRESIDENT. Is there any further discussion?

Mr. HUSTON. I am quite a little interested in the method which Mr. Macfarlane has referred to with ammonium chlorid. It recalls my troubles of some years ago, for we have done a good deal of work with these materials in our laboratory. While I think it is quite right that the association should give reasonable attention to the request of a manufacturer of a product which does not seem to fulfill the manufacturer's expectations when tried by our methods, I am convinced that the Wagner method is utterly without value for working basic slag. Any reagent the curve of whose solvent action will go up rapidly and then return to zero is worthless for this purpose. With the Wagner reagent there is an enormous temperature range, and a very great difference in the amount dissolved according to the time involved. If you undertake to remove the free lime with ammonium chlorid you take out every element in the slag but phosphate. You can tear slag to pieces if you treat it with ammonium chlorid long enough. I much prefer sugar solution, as I think it disturbs the slag much less. I think we are putting ourselves to a needless trouble in trying to determine availability in basic slag. That is simply a question of total phosphoric acid and degree of fineness.

Mr. WHEELER. A large number of tests have shown very strong evidence that a 2 per cent citric-acid solution does give results according very closely with plant tests. It seems necessary to have immediately some method for the analysis of basic slag, and I think it would not be an unsafe policy to adopt the 2 per cent citric-acid solution until such time as the association can assure itself of a better procedure.

Would it be contrary to the constitution to adopt any method of analysis tentatively? Could this method be adopted as a provisional method for basic slag? Would it be constitutional?

The President (after reading section 7 of the constitution). I think it could be done constitutionally. If we had a method already we could not; but as we have none, we can adopt one.

Mr. Wheeler. I am interested in this matter because basic slag is of great importance in our agriculture. Some shipments were made to Rhode Island this year, and at any minute the goods are likely to be put on sale and it seems to me we must meet this problem at once. I think the gentlemen here are perfectly familiar with the fact that careful and extended tests have been made in Europe and we know the method adopted by the German chemists does agree very closely with plant experiments. I move that we adopt some method to use with basic slags temporarily. I move the adoption of the 2 per cent citric-acid solution method as a provisional method until such time as we are in a position to adopt a method as official.

Mr. Magruder. I second Mr. Wheeler's motion. In Virginia we have basic slags to analyze, and I don't know what to do with them. I would like the association to adopt some method for them.

Mr. Frear. I have experienced something of the same difficulty with this matter, and I shall be very glad to second the motion of Mr. Wheeler.

Mr. Dyer. Perhaps you would like to know what we do on our side in regard to this matter. We have no official processes of fertilizer analyses in England, and in the matter of basic slags the commercial custom is to determine only the total phosphoric acid and take into account the degree of fineness. These two things together are taken as the measure of value. It is within my own personal knowledge that there are now on the market varieties of slag which are the result of a somewhat different process of steel making and having such variation that some have almost twice the availability of others. Mr. Macfarlane mentioned that when he was over there he referred this matter to our Society of Public Analysts, of which I have the honor to be president, and a committee was appointed to consider matters which Mr. Macfarlane brought before us. The committee consisted of those members who are more especially connected with fertilizer analysis. That committee made a report to the council of the society shortly before I sailed, and the council meeting at which this report was presented has taken place since I left England. I presume this report will be adopted and I think it will be interesting if I read to you the report of the committee on the question:

Resolution of the subcommittee appointed by the council of the Society of Public Analysts, London, England, which met on October 16, 1900, to consider Mr. Macfarlane's proposal relative to the analysis of fertilizers.

The committee have very carefully considered Mr. Macfarlane's letter and the documents therein referred to, but do not see their way to recommend the council to take steps in the direction either of laying down standards or of prescribing arbitrary methods for the analysis of fertilizers.

With regard to the subject more especially dealt with in Mr. Macfarlane's report (Bulletin No. 70), the committee do not make any recommendation in regard to the formulation of an official process for the estimation of available phosphoric acid in basic slag or other fertilizers. The committee, however, are unanimously of opinion that the ammonium citrate process, which is at present officially used in the United States of America, while affording useful means for the proximate determination of reverted phosphate in superphosphate, dissolved bones, and similar acid manures, is, nevertheless, in no sense an adequate means of measuring the amount of available phosphate that does not happen to have gone through the process of solution and reversion.

The committee are further of opinion that the citrate of ammonium process is wholly inapplicable to the analysis of basic slag, and that if any process of analysis is to be used for distinguishing between total and available phosphate in that manure it must be an acid process and one proceeding on some such lines as the present process of Professor Wagner.

This is a very cautious resolution, but it covers what is virtually a recommendation.

Mr. Hills. I have not followed the discussion very closely, but there is one point about which I would like to speak. I believe our State laws require that the reverted phosphoric acid be dissolved with ammonium citrate.

Mr. Wheeler. Is there more than one State law in the Union that requires the determination to be made at a given temperature?

Mr. Carpenter. I don't think it is generally recognized that basic slag is as valuable as acid phosphate, but if this acid test is applied, as I understand it, the result is that it is put on the same basis as acid phosphate. The results on some soils of basic slag may prove as beneficial as those produced by using acid phosphate, but on other soils the results with basic slag are of hardly any value at all, and it seems unjust to manufacturers of acid phosphate to put an article of this sort on the same basis.

Mr. Myers. As I understand it, Mr. Huston struck the nail on the head when he said that the total phosphoric acid and the degree of fineness had a great deal to do with the question. You are dealing with a special compound in this case which is almost as distinct from a manufactured acid phosphate as sulphate of potash is. The question is whether there is in that compound any of the material which we call available phosphoric acid in the sense in which we use that term in describing an acid phosphate. I doubt exceedingly whether this is the case, but plant and pot experiments have demonstrated beyond doubt that when this material is mixed with the soil it does bring results, and the action to make a tentative method of analysis for these goods seems to me to be an exceedingly wise one. Those gentlemen who have absolute laws regulating the methods of analysis in their States can apply them in such way as they see fit, but as a general preparation for the whole country it seems to me that there should be some tentative process for the determination of its constituents.

Mr. FREAR. When the day comes, and it seems to be coming now, that basic slag is brought into our land, it seems to me it would be wiser for us to follow those methods of analysis which have been adopted as the result of long experience abroad, so that our results will be comparable. In that way I think we will be able to avoid controversy with importers and manufacturers and dealers on the other side.

Mr. DYER. Though we have no official process at home, I often have to make analyses of slag sold on the Continent, and I can say of the Wagner process that it gives concordant results if worked with any reasonable degree of care, which is a good deal to say of an arbitrary process which dissolves only a fractional part of the substance. Different operators have not the slightest difficulty in getting concordant results.

Mr. MACFARLANE. Since the opinion of this meeting seems to be that Wagner's method should be adopted, I would like to ask if the gentlemen have considered the possible consequences. Suppose a manufacturer of superphosphate finds that this material is imported into your country and incorporated with a certain amount of phosphoric acid, and he finds also that you use this 2 per cent solution, and at the same time you apply a totally different solution to the insoluble part of his fertilizer. I think he is very likely to make objection. I know it to be the case that the fertilizer manufacturers in Canada would make very decided objections. I think it would justly cause controversy if you applied the Wagner system to the basic slag and not to other fertilizers. I have no doubt that Wagner's experiments were thorough and correct, and that in his agricultural experiments and experiments in his laboratory and garden he obtained results which justify him in saying that the proper solution is a 2 per cent solution. It seems to me that it is very necessary that you should be able to say to manufacturers, "You all get the same treatment." I would like to impress upon you the necessity of regarding the subject from the point of view of the manufacturers.

Mr. WHEELER. I ought to have stated that my motion was not made with the idea of putting off consideration of the paper presented by Mr. Macfarlane, because I had intended to move after this motion that the paper be referred to our committee on recommendations and be given due consideration with the idea that its suggestions might be taken up and tried by our association. But in the meantime we ought to have some method, and it seems to me that the 2 per cent solution is that one.

The PRESIDENT. You suggest that the paper be referred to the committee on recommendations. Would it be entirely proper that your motion should go to that committee also or to a special committee?

Mr. WHEELER. I would move that the motion to adopt the 2 per cent citric acid solution, and also the consideration of the paper, be be referred to our committee on recommendations.

Motion seconded.

Mr. BARTLETT. Is not this going to give a chance to the manufacturers of bone meal, and other kinds of raw phosphates, to demand that this same method be used on their goods? It seems to me they could make that demand, and that until we know what method should be used in this country it would be better to act upon the suggestion of Mr. Huston and consider the fineness of the material and the total phosphoric acid it contains.

The motion of Mr. Wheeler was carried.

Mr. Williams then presented a paper as follows:

KILGORE'S MODIFICATION OF THE VOLUMETRIC METHOD OF ESTIMATING PHOSPHORIC ACID.

By C. B. WILLIAMS.

In the laboratories of fertilizer control stations and other institutions where a large number of determinations of phosphoric acid are required to be made quickly and accurately each year, it has been recognized for some time as almost imperative that some method shorter than the "gravimetric" should be devised. In 1894, Mr. Kilgore, then reporter on phosphoric acid for the Association of Official Agricultural Chemists, realizing this urgent demand, was the first to take up systematically the task by first thoroughly investigating the modified volumetric method himself, and then submitting it, as reporter, to the test of the association. In his report he gave credit to Mr. Henry Pemberton, who had the previous year published the description of a method based on this principle and which had been used very satisfactorily by a number of chemists. With this latter method Mr. Kilgore had obtained fairly good, but not uniformly good, results.

After considerable experimentation as regards precipitant, precipitation, and filtration, he proposed a modification of this method, stating that he had found that the modification gave him more satisfactory results with fertilizers. Since then each successive year has witnessed a still further modification, until now we have a method that is used in a large number of laboratories in America, with very gratifying results both in point of accuracy and rapidity.

By the adoption of the shaking method the writer was enabled, during the past spring, to precipitate, wash, and titrate, according to the modified volumetric method, 30 phosphoric-acid samples daily. The reagents used are the same as those prescribed for the volumetric method by the Association of Official Agricultural Chemists, except that the strengths of the standard solutions of potassium hydroxid[1] and nitric acid are made up so that 1 cc of each will represent 0.5 milligram of phosphoric acid, this being one-half the strength given in the association method.

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[1] The standard potassium hydroxid is rid of CO_2 by first dissolving it in 95 per cent alcohol, letting settle, and then filtering off by reverse filtration the supernatant solution free from the insoluble potassium carbonate. This method is much quicker and simpler than the barium hydroxid method.

The method as carried out is as follows:

Total phosphoric acid is brought in solution in the usual way by boiling in a 200 cc flask, on a sand bath, 2 grams of fertilizer with 30 cc of concentrated nitric acid and 10 cc of concentrated hydrochloric acid to about 8 or 10 cc concentration; except in fertilizers containing much iron and alumina, in which instance 30 cc of concentrated hydrochloric acid alone is first added and boiled for about 30 or 40 minutes; then, after slightly cooling, 30 cc of concentrated nitric acid is added and the boiling continued until the excess of hydrochloric acid is removed. After cooling, make up to volume and take an aliquot part after filtration, or allow to stand several hours before measuring out. The latter is done in order that the supernatant liquid may become perfectly clear so it can be measured out with a pipette without filtration.

Twenty cc of solution, corresponding to 0.2 gram of fertilizer (except in samples containing over 20 per cent of phosphoric acid, when 10 cc is used), are measured into a 500 cc Erlenmeyer flask, the inside diameter of whose neck measures about 40 millimeters, and to it is added 10 to 12 grams of ammonium nitrate and 50 cc of distilled water. Neutralize the excess of acid with ammonia. When the contents have cooled, 30 cc of recently filtered molybdic solution are added and the flask, securely stopped with a rubber stopper, is placed in a Wagner shaking machine, which is revolved by a hot-air motor, and here shaken for 30 minutes. The shaking machine is maintained at 45 to 55 revolutions per minute, as this velocity has been found to give the maximum agitating efficiency. Remove flask from shaking machine and filter and wash by suction on a filter prepared as follows:

Through the rubber stopper in a 16-ounce pressure bottle of Erlenmeyer form is passed the small end of a carbon filter. In the bottom of this is a perforated porcelain plate or disk, to which is rigidly fastened a No. 19 copper wire, about 25 centimeters long, that projects downward into the pressure bottle. The disk is covered with a thin layer of asbestus.

After thoroughly transferring the ammonium phosphomolybdate and washing out the flask on to the asbestos filter, six more washings are given the precipitate. Then remove the stopper from the pressure flask with the small end of the carbon filter still stuck through it and hold upright over the sink and wash the outside free from acid with distilled water. Reverse the carbon filter into the mouth of the flask that originally contained the precipitate, still holding the small stem, and by means of the copper wire that extends beyond the small end of the carbon filter push out the disk, asbestos, and precipitate into the flask; wash disk and inside of carbon filter carefully and titrate, using a stirring rod about 30 centimeters long to thoroughly agitate during the operation.

In determining insolubles, 40 cc of solution, corresponding to 0.4 gram fertilizer, are taken. The precipitation, shaking, washing, and titrating are practically the same as with totals, except that little or no water is added in preparing for precipitation.

During the past spring 1,000 totals and 1,000 insolubles were made by the above method with not a single incomplete precipitation, the yellow precipitate always coming down in a granular form that was easily filtered and washed. Distilled water must be used in washing, as the suspended matter in ordinary water, in case it contains any, will not only retard filtration, but will form a compact coating over the precipitate that will greatly increase the difficulty of effecting a solution of the yellow precipitate with standard alkali as well as obscuring the color change of the indicator.

During the past year a large number of comparative results to test the volumetric method, as described above, with the regular official gravimetric method of the association, have been obtained in the laboratory of the division of chemistry, North Carolina department of agriculture, on commercial fertilizers offered for sale in the State. In all instances results were extremely satisfactory.

During the past summer three samples (two of ground phosphate rock and one of American slag) sent out by the referee on phosphoric acid for the Association of Official Agricultural Chemists were analyzed, with the following results:

Comparative determinations of phosphoric acid by gravimetric and volumetric methods.

No.	Gravimetric method.	Volumetric method.	No.	Gravimetric method.	Volumetric method.
	Per ct. P_2O_5.	*Per ct.* P_2O_5.		*Per ct.* P_2O_5.	*Per ct.* P_2O_5.
389	13.55	13.45	390	17.35	16.90
	13.43	13.50		17.33	....
	....	13.51		17.27	....
	....	13.50	391	26.01	25.90
390	17.21	16.88		26.10	25.80
	17.23	16.93		26.11	25.85
	17.27	16.95		26.02	25.88

It will be noticed that the gravimetric results on sample No. 390 are perceptibly higher than those determined volumetrically. This is most probably due to the presence of iron in the magnesium pyrophosphate as a qualitative test, for iron revealed its presence there. As sample No. 391 contains 4.70 per cent of ferric oxid, this may account for the wider variation than in No. 389 between the gravimetric and volumetric results.

Mr. BARTLETT. Before leaving the subject of phosphoric acid, I would like to say a word. We were unable to cooperate in this work during the past year because of the pressure of other matters. By a strange coincidence, the work in which we were engaged, namely, the determination of alumina in baking powder, is found to bear directly on the subject under discussion. This may seem strange, but as a matter of fact many of the baking powders on the market contain the same mineral ingredients as basic slags, although in different proportions. The percentage of alumina is the same, ranging from 3 to 6 per cent. The method which we have used, and quite successfully, is the acetate method. I would suggest that while this matter of the determination of alumina, iron, phosphoric acid, and lime is being considered in the case of fertilizer materials it might be well to consider the same bodies as found in baking powders. If these two matters can be taken up together I think the work of the association would be facilitated. The acetate method, in a somewhat different form, has given results which agree closely with the old standard method. This may be out of order, but I do not think it would be in order in connection with the examination of food products.

After some discussion as to the time of holding the next session, the meeting adjourned to meet at 9 o'clock the following morning.

SECOND DAY.

SATURDAY—MORNING SESSION.

The meeting was called to order at 9 a. m., with the president, Mr. Kilgore, in the chair.

The report on sugar was then called for, and the following letter from the absent referee was read:

San Francisco, Cal.,
October 25, 1900.

Dr. H. W. Wiley,
Secretary Association of Official Agricultural Chemists,
Washington, D. C.

Dear Sir: Owing to my departure for the Hawaiian Islands, I regret to inform you that I have not been able to complete the report of the referee on sugar in time for the meeting of the association in November. The work preliminary to my leaving the sugar experiment station has so occupied my time as to render it impossible to finish my report as referee.

The amount of cooperation received in this work has been very small, and only two reports have been received, one from Dr. John White and the other from Mr. R. B. Hellner, both of Nebraska. These, together with the work done at the sugar experiment station in Louisiana, are all the data at hand, and from this very few conclusions can be drawn. I hope that the absence of the report this year will not detract from the association work.

I also sincerely regret not being able to comply with your request regarding the revision of Bulletin No. 46, but the same reasons apply in this instance.

Again assuring you of my regrets, etc., and hoping for a successful and instructive session of the association, I am,

Yours very truly,
R. E. Blouin,
Referee on Sugar.

Mr. Wiley. I thought the members might be interested in the brief report of the international committee for the unification of methods of sugar analysis at the third international congress of applied chemistry, which was held in Paris in August, 1900. This committee was appointed by the international congress of applied chemistry at the Paris meeting in 1896. The first meeting, which I did not attend, was held at Braunschweig. The second meeting was held in Vienna, in 1898, with about thirty members of the committee and invited guests present, under the presidency of Dr. Herzfeld, of Berlin. Twelve members of the committee were present at the Paris meeting, also under the presidency of Dr. Herzfeld.

The chief points discussed were the influence of temperature upon the specific rotation of sugar and the adoption of general principles of analysis for international guidance.

After a prolonged discussion the committee voted to recommend that all polariscopes be standardized for the usual temperature at which they were employed, the committee recognizing the fact that variations in temperature caused marked changes in specific rotation. This principle is one which has been put in practical operation in this country, in official work, for more than ten years. It has also been recognized in the British West Indies for a longer period and proper corrections made in the polarizations for the high temperatures which there prevail.

The specific rotation of sugar decreases as the temperature rises, while the specific rotation of quartz increases with a rising temperature. Since in ordinary instruments the compensation for the sugar solution is secured by means of a quartz wedge, a less thickness of it is required for a given degree of rotation at a high temperature than at a lower one. Thus the reading of the scale, which is attached to the quartz wedge, is lower than it really should be. At the same time the specific rotation of the sugar diminishes, and thus adds to the error.

Most careful measurements of the magnitude of these errors, on quartz compensating instruments, have shown that they amount to about one-tenth of a per cent of sugar on the sugar scale for each rise of 3° of temperature. Since the average temperature of polarizations in this country is nearly 10° higher than the temperature at which our standard instruments are graduated, viz, 17.5°, it is seen that the average magnitude of the error in this country would be almost three-tenths of 1 per cent.

The importance of allowing for this error, both in commercial valuations and in the classification of sugars for customs duties, is at once apparent. It is gratifying to know that the international committee on the unification of sugar analysis has accepted the principle of correction which has been adopted for so many years in the official work of this country.

In experiments conducted in my own laboratory I demonstrated beyond doubt the magnitude of these errors, and the paper containing the results of my investigations was presented at the meeting of the international congress of applied chemistry and also published in the Journal of the American Chemical Society for July, 1899.

Mr. Fraps. I would like to ask a question as to the temperature at which these experiments were carried out, and what kind of sugars were used.

Mr. Wiley. The highest temperature which I used in my work was 40° and the lowest was zero.

Mr. Fraps. What kind of sugar did you use?

Mr. Wiley. Chemically pure sugar.

The report on soils was then presented by Mr. Hartwell.

REPORT ON SOILS.

By Burt L. Hartwell, *Referee.*

As the analytical data included in the present report represent the work of two years, it may not be out of place to review briefly what has been done since my appointment as referee on soil analysis in 1898.

As soon as possible after that time a circular letter, published in our last proceedings, was sent to the Experiment Station chemists. This letter stated the recommendations made by this association at the meeting held here two years ago regarding the soil work. They were (1) a trial of the so-called international method for determining assimilable potash, i. e., using dilute nitric acid as a solvent; (2) a further trial of Hollemann's method for the determination of the active lime compounds, i. e., using water saturated with carbonic acid, and (3) further tests with alkaline ammonium chlorid as a solvent for potash. An appeal was made for suitable soils for carrying on the work of testing solvents which should serve for the determination of the more active soil constituents. In another paper will be presented the results of some preliminary work upon the soils received in response to this request. Suggestions concerning the work and cooperation were also urged.

It was deemed advisable, on account of the immediate need for samples, to distribute to those who expressed a willingness to take part in the investigations certain soils upon which considerable work had already been done by this association, and in addition two soils from California. It has been thought best to present in this connection such information as it has been possible to obtain concerning the soils employed.

Description of the Soils.

No. 1, from Junction City, Boyle County, Ky., farm of Thomas R. Walker.—From the same lot of soil as was distributed as No. 1 by the referee for 1898. A light clayey loam, cleared from original timber about twenty-five years ago. Probably of the Devonian formation. Surface soil 4 to 6 inches deep. Below this is a yellow clay subsoil mixed with gravel, 2 to 4 feet in depth, and under this latter a blue clay.

Field experiments were conducted under the direction of the Kentucky Experiment Station upon land which this sample represents, the experiment with potatoes being upon the same field from which the sample was taken, and those with maize and beans upon the same kind of land. Below are given the yields per acre:

Yield per acre of potatoes and ear corn, 1895–'96.

No. of plat.	Fertilizers supplied.	1895, potatoes.	1896, potatoes.	1896, ear corn.
		Bushels.	*Bushels.*	*Bushels.*
1, 5, 10, av	None	78	46	14
2	N	72	45	10
3	P_2O_5	122	155	25
4	K_2O	77	55	14
6	N, P_2O_5	146	208	37
7	N, K_2O	75	71	15
8	P_2O_5, K_2O	125	171	28
9	N, P_2O_5, K_2O	121	284	27

The fertilization per acre was 160 pounds of nitrate of soda, 140 pounds of double superphosphate, and 160 pounds of sulphate of potash.

Navy beans, 1896.

Fertilization.	Crop in pounds.
None	909
750 pounds Orchilla guano	1,134
750 pounds acid phosphate	1,620

The sample was taken to the depth of 4 to 6 inches, in March, 1898, from many places. The land had received an application of 200 pounds of acid phosphate per acre a few months before. It is impossible to say how much influence this application would have upon the amount of phosphoric acid removed by weak acids, but even if all of the available phosphoric acid in the acid phosphate should be removed by the weak acid solvents, which is not at all probable, the percentage would not be increased 0.003 per cent. The entire amount of phosphoric acid actually found by treatment with N/5 hydrochloric acid within a year after the application of acid phosphate was made amounted to only 0.0026 per cent.

Both crop records and analysis showed this soil to be deficient in assimilable phosphoric acid, and it was again distributed in 1899 chiefly for the determination of the amount of phosphoric acid soluble in N/5 nitric acid. This solvent was recommended for trial by the association for determining active potash, and it was thought desirable to ascertain if its action upon the phosphoric acid of the soil were similar to that of N/5 hydrochloric acid, which had been adapted provisionally; if so, a weak solution of nitric acid may be applicable for the determination of the more active forms of both phosphoric acid and potash, and thus make the use of two solutions unnecessary.

No. 2. From the Kentucky Agricultural Experiment Station.—From the same lot of soil distributed as No. 2 by the referee for 1898. The soil was taken in March, 1898, from acre P., plat 7, at the depth of 5 or 6 inches. The land had never received any fertilizer, and experiments carried on for a number of years indicate a marked deficiency in available potash.

A description of this soil, the crop records, and analytical results heretofore obtained upon the same by this association are given in my last report.[1] This soil was distributed for the estimation of potash soluble in N/5 nitric acid and in alkaline ammonium chlorid.

No. 3. From the Rhode Island Agricultural Experiment Station.—This soil was sampled to the depth of 4 inches in the fall of 1894 from the unfertilized plot of a soil test. Samples collected at the same time from plots 2 and 5 of this experiment were used by the referee for 1895. A description of the soil, crop results, and analytical data may be found in the proceedings for 1899 (p. 81). The soil needs liming or other alkaline treatment to insure a good yield of most crops. It was distributed for the determination of lime soluble in N/10 acetic acid and in water saturated with carbon dioxid.

No. 4. From Ventura County, Cal.—Orange orchard of N. B. Blanchard near Santa Paula; a representative of the arid upland or bench soils. A silty soil of light umber color when dry, becoming blackish when wet; very easily tilled, retaining its tilth remarkably, so that an ax handle can be thrust perpendicularly into it the entire length with little exertion. In the lower bench of the valley where the sample was taken the material remains apparently the same to a depth of from 12 to 20 feet; toward the hills there is a second bench where the soil is apparently the same, but of a slightly reddish tint; on the mountain slopes the soil, still quite similar in its working qualities, is of a decidedly reddish tint and is remarkable for its retention of natural moisture, enabling it to produce corn without irrigation. This soil was distributed as one containing plenty of plant food, and the various determinations by the use of weak solvents were requested for comparison with results obtained on the deficient soils.

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[1] Proceedings A. O. A. C., 1899, p. 78.

No. 5. From the Sierra Foothill Station, near Jackson, Amador County, Cal.—Sample taken to the depth of 12 inches from about half way down the central hill; an orange-red loam, the lumps of which are easily crushed between the fingers when dry, and show considerable coarse sand; when wet it becomes only moderately adhesive, while its color darkens materially; slate fragments are intermingled more or less, much of the sand being comminuted slate. The natural growth[1] characteristic of this soil includes all the trees and shrubs of the foothills at large, so that as far as the vegetation can indicate, this red soil is representative of the higher class of land on which the poison oak and toyon alone are found in the uplands. This soil contains a high percentage of calcium and potassium oxids, which render soils of this character very productive for a while, even though the amount of phosphoric acid is small. Farmers have, however, after a time found it to their advantage to supply phosphoric acid. It was thought desirable to test the solvents upon this soil, which is representative of so large a class.

Directions for the work to be undertaken upon the above-mentioned soils were distributed in March, 1899. It was possible to secure so few results before the meeting of the Association of Official Agricultural Chemists at San Francisco in July, 1899, that the cooperating chemists were requested to continue the work with the same soils and directions during the present year. These directions were unexpectedly published in connection with my last report. They contain a few mistakes which should be noted, viz, next to the last line in section 2 reads, "and 5 cc strong nitric acid," the word "and" should be replaced by "add;" soil No. 5 was published as coming from Arlington Heights, southern California. This could not be obtained in time, and a soil from the Foothill Station, as previously mentioned, was finally employed as No. 5.

MECHANICAL ANALYSES.

Mechanical analyses of soils from plats in close proximity to those from which Nos. 2 and 3 were taken may be found on pages 79 and 81, respectively, of the proceedings for 1899. Below are given results furnished by Loughridge, of Berkeley, Cal., which represent the mechanical condition of the soils from the localities where Nos. 4 and 5 were taken.

Mechanical condition of soils from same localities as samples Nos. 4 and 5.

Soil.	Similar to No. 4.	Similar to No. 5.
	Per cent.	*Per cent.*
Gravel over 1.2 mm diameter	....	13.95
Gravel between 1.2 and 1 mm diameter	....	.20
Coarse sand between 1 and 0.6 mm diameter	3.53	5.85
Fine earth less than 0.6 mm diameter	96.47	80.00
	100.00	100.00

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[1] Cal. Agr. Expt. Sta. Rpt., 1888 and 1889, p. 79.

Hydraulic values of the fine earth borne up by water rising at the velocities indicated in millimeters per second.

Constituents of fine earths.	Hydraulic values.	No. 4.	No. 5.
	mm.	*Per cent.*	*Per cent.*
Sand	64	4.11	2.17
	32	6.45	2.89
	16	7.01	5.04
	8	11.48	4.03
	4	16.36	5.91
Silt	2	12.67	8.21
	1	8.03	7.68
	.5	5.14	9.96
	.25	1.20	4.64
	.25	14.04	30.26
Colloid clay	.0023	15.02	16.43
		101.51	97.22
Hygroscopic moisture in fine earth		5.49	6.00
Water-holding capacity		32.10	47.50

Soils Nos. 1 and 2 had been passed through a sieve approximately 1 mm in diameter, as was the case in 1898; No. 3 through a sieve about 2 mm in diameter; Nos. 4 and 5 through a 0.6 mm sieve.

Below are given the data furnished by A. M. Peter, of Kentucky, concerning the coarse soil of the lots from which Nos. 1 and 2 were taken:

Mechanical condition of coarse portion of soils from which samples Nos. 1 and 2 were taken.

Material.	No. 1.	No. 2.
	Per cent.	*Per cent.*
Larger than 3 mm sieve	1.69	0.67
Between 2 and 3 mm sieve	.73	1.05
Between 1 and 2 mm sieve	1.26	4.21
Between one-half and 1 mm sieve	.10	1.03

Soil No. 3 contained 95.55 per cent of material finer than 2 mm, 1.48 per cent between 2 and 4 mm, and 2.97 per cent coarser than 4 mm.

RELATIVE WEIGHTS OF THE SOILS.

The "apparent specific gravity" of Nos. 1 and 2 were obtained by Mr. Peter by packing the soil into a receptacle as tightly as possible, by jarring, and comparing the weight with that of an equal volume of water. The specific gravity for No. 1, with 1.01 per cent of moisture, was found to be 1.27 in the sifted and 1.23 in the unsifted soil; that for No. 2, with 2.16 per cent of moisture, was 1.25 for the sifted and 1.20 for the unsifted soil. Soil No. 3, air dried, when well shaken down, weighed 1.21 times as much as the same volume of water. Similar determinations were made upon the air-dried samples, Nos. 4 and 5, before sifting; the specific gravity of No. 4, with 2.14 per cent moisture, was 1.35, and of No. 5, with 1.12 per cent moisture, 1.19.

Although these data are not as complete in all cases as they should be, they enable one to calculate roughly the number of pounds of the dry soil which would be contained in an acre of land to the depths sampled. The number of pounds per acre in round figures is thus found to be for No. 1, at 6 inches deep, 1,590,000; for No. 2, at

6 inches deep, 1,500,000; for No. 3, at 4 inches deep, 1,010,000; for No. 4, at 12 inches deep, 3,470,000; for No. 5, at 12 inches deep, 2,560,000. These figures enable anyone to calculate roughly the amount of assimilable plant food per acre at the various depths from the percentages of the same which are found by analysis.

It must be plain to the members of this association that our percentage results upon agricultural soils of different depths should not be expected necessarily to be in direct relation to the crop-producing qualities of the same. A comparison of the amounts of a given ingredient found by analysis to exist in an assimilable form within a given area at the depth to which the roots penetrate would seem to be more reasonable. This necessitates a consideration of the depth to which the roots of the same kind of plant will seek plant food under the existence of different conditions. In making comparisons of the percentages of given ingredients found in the soils employed in the work of the present year these considerations should not be overlooked, particularly as there will be occasion to compare shallow soils from humid sections with deep soils subjected to different conditions as to rainfall.

COMPLETE ANALYSES.

The following determinations were made by Mr. McGuigan, Agricultural College, North Dakota.

Determinations made with samples Nos. 1, 2, 3, 4, and 5.

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Moisture	0.69	1.55	1.24	1.10	0.76
Volatile matter	5.26	7.66	9.54	2.83	5.71
Total silica (SiO_2)	87.49	78.40	81.51	87.39	78.15
Iron (Fe_2O_3)	4.50	8.00	4.10	3.58	5.64
Alumina (Al_2O_3)	0.87	2.39	2.01	2.44	7.88
Phosphoric acid (P_2O_5)	0.17	0.35	0.13	0.21	0.12
Manganese (MnO)	....	....	0.08	0.09	....
Lime (CaO)	0.25	0.46	0.34	0.71	0.48
Magnesia (MgO)	0.16	0.23	0.08	0.10	0.27
Sulphuric acid (SO_3)	....	....	0.29	0.12	0.07
Potash (K_2O)	0.17	0.23	0.21	0.38	0.46
Soda (Na_2O)	0.20	0.24	0.27	0.34	0.32
Total	99.76	99.51	99.80	99.29	99.86
Nitrogen	0.12	0.17	0.236	0.0623	0.07
Extractives (Humates)	4.98	7.86	5.76	0.85	....
Humus	2.00	3.02	4.34	0.22	....
Alkalies	0.113	0.345	0.546	....	....
Phosphoric acid	0.026	0.062	0.036	....	....

The following statements from Mr. Ladd, of North Dakota, accompanying the above analyses, are explanatory of certain determinations, viz: "I have termed humates the entire extractives dissolved out by the ammonia solution after the soil has been treated with weak hydrochloric acid; as found by evaporating an aliquot portion of the extract. The phosphoric acid and alkalies reported are those found in this residue. The alkalies in the humus extract are weighed as chlorids."

POTASH.

While N/5 hydrochloric acid has been accepted provisionally as a solvent for assimilable phosphoric acid, we have not found any solvent for potash which has proved satisfactory in the minds of the referees. Recommendations to discontinue the use of citric acid, calcium chlorid, and ammonium chlorid have been made by different referees. Further trial with the last solvent, however, was urged, and a method heretofore untried by the association recommended, viz, the so-called international method for available potash, in which dilute nitric acid is used. At the congress of chemists held in Paris in 1889 it was pointed out that with a 0.05 to 0.1 per cent nitric-acid solution, the quantity of potash which goes into solution increases by continued stirring of the mixture with the time of action of the acid up to a certain maximum which is reached in from three to four hours, and after that it is not changed even when the strength of the acid mixture is increased to 2 per cent.[1] If this be true a decided advantage at once appears in the use of dilute nitric acid. N/5 nitric acid was the strength determined upon by your referee that we might have a solution of definite normality and that the results obtained by the same solvent upon phosphoric acid would be comparable with those by N/5 hydrochloric acid, which has been adopted provisionally.

Thorn Smith stated in a letter accompanying his results that he lacked confidence in them because of constant unavoidable interruptions, in consequence of which the directions in many cases were not followed as to shaking and time of digestion.

C. C. Moore, instead of shaking the solution every half hour, as stated in the directions, used a continuous shaking machine. Having recorded these exceptions to the regular method as outlined, it has been thought best to include the results by Smith and Moore with all other results which follow without further comment. All of the succeeding results represent percentages of water-free soil.

Determination of potassium oxid in soil samples.

Analyst.	Solvent. *a*	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.	Soil No. 5.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
G. S. Fraps, Raleigh, N. C	$N/5HNO_3$	....	.0063	....	....	....
Thorn Smith, Moscow, Idaho		....	.0060	....	.0295	.0195
R. J. Davidson and W. E. Ellett, Blacksburg. Va.		....	.0087	....	....	.0178
J. A. Bizzell, Raleigh, N. C		....	....	....	.0143	.0157 .0125
C. C. Moore, Division of Chemistry, Washington, D. C.		.0190	.0163	.0140	.0358	....
M. E. Jaffa, Berkeley, Cal	$N/5HCl$	.020	.016	.019	.036	.029
Do	2 per cent citric acid. *b*	.010	.008	.012	.018	.013
Thorn Smith, Moscow, Idaho	Alkaline $(NH_4)Cl$.	.0122	....	....	....	....
Davidson and Ellett, Blacksburg, Va.		....	.0075	....	....	....
J. A. Bizzell, Raleigh, N. C		....	....	....	.0151	.0139

a In the acid solvents allowance was made for the neutralizing action of the soil, so that the strengths at the end of the digestion are given.

b Fifty gms. of soil digested at room temperature in 300 cc of solution for twenty-four hours with frequent shaking.

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[1] Wiley's Principles and Practice of Agricultural Analysis, p. 381.

The comparative yields from certain plats of field soil tests which furnished data as to the amount of assimilable potash.

The first three terms of proportions, the last terms of which are given in the following columns.	Soil No. 1, potatoes. Sampled in Mar., 1898.		Soil No. 2, corn on the ear. Sampled in Mar., 1898.		Soil No. 3, corn and stover. Sampled in fall of 1894.	
	1885.	1896.	1889–92.	1894–97.	1890, 1891.	1892, 1893.
Nothing plat : K_2O plat : : 1 :	0.9	1.2	1.8	1.9	....	....
N plat : N, K_2O plat : : 1 :	1.0	1.6	2.0	1.8	1.6	1.7
P_2O_5 plat : P_2O_5, K_2O plat : : 1 :	1.0	1.1	1.9	1.9	1.3	1.4
N, P_2O_5 plat : N, P_2O_5, K_2O plat : : 1 :	0.8	1.4	2.0	2.1	....	....

NOTE.—It will be evident from the above table that numbers greater than 1 show an increased yield from the use of potash, while those equal to or less than 1 show no increase.

According to the above table of comparative yields on the deficient soils, Nos. 1, 2, and 3, one would attribute the greatest deficiency of assimilable potash to No. 2; it is more uncertain, however, considering the different crops employed, as to whether No. 1 or No. 3 ranks next in deficiency. In view of the fact that there was no deficiency of assimilable potash in soil No. 1 during 1895, whereas there was a lack in soil No. 3 from the very first, it may be justifiable to conclude that the latter is more deficient. If these ideas be correct regarding the relative deficiencies of the three soils, then Jaffa's results with N/5 hydrochloric acid are in accord. The results by Moore with N/5 nitric acid are not in accord, because the least potash was not obtained from soil No. 2, which apparently was the most deficient. It will be noticed that no two of the acids agree concerning the relative amounts of assimilable potash in soils Nos. 1, 2, and 3 in the cases where comparisons have been possible. No conclusions, however, should be drawn from so few determinations, particularly when ideas concerning relative crop-producing powers are so liable to error.

All of the solvents employed for assimilable potash dissolved less potash from the deficient soils than from the fertile California soils. The same is seen to be true also of strong hydrochloric acid by glancing at the results by Mr. McGuigan given previously. The fact that only 0.001 per cent more of potash was extracted by 2 per cent citric acid from soil No. 5, which is supposed to be rich in assimilable potash, than from the deficient soil No. 3 should not be overlooked.

PHOSPHORIC ACID.

Phosphoric acid extracted by N/5 hydrochloric and nitric acids.

Analyst.	Solvent.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.	Soil No. 5.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
G. S. Fraps, Raleigh, N. C.	N/5 HCl	0.0026	....	....	....	....
	N/5 HNO_3	.0029	....	....	....	....
J. A. Bizzell, Raleigh, N. C.	N/5 HCl	....	....	....	....	....
	N/5 HNO_3	....	....	....	0.1341	0.0020
Thorn Smith, Moscow, Idaho	N/5 HCl	....	....	....	.1422	.0011
	N/5 HNO_3	.0029	....	....	.1321	.0018
Davidson and Ellett, Blacksburg, Va	N/5 HCl	....	....	0.0284	....	Tr.
	N/5 HNO_3	.0019	....	.0186	....	Tr.
C. C. Moore, Division of Chemistry, Washington, D. C	N/5 HCl	.0016	0.0666	.0265	.1350	....
	N/5 HNO_3	.0010	.0854	.0190	.1737	....

Comparative yields from certain plats of field soil tests which furnished data as to the amount of assimilable phosphoric acid.

The first three terms of proportions, the last terms of which are given in the following columns:	Soil No. 1, potatoes sampled in Mar., 1898.		Soil No. 2, corn on the ear sampled in Mar., 1898.		Soil No. 3, corn and stover sampled in fall of 1894.		
					Unlimed.		Limed in 1896.[1]
	1895.	1896.	1889 to 1892.	1894 to 1897.	1890 and 1891.	1892 and 1893.	1898 to 1900.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Nothing plat: P_2O_5 plat : : 1 :	1.6	3.4	0.9	1.0	....	....	....
N plat: N, P_2O_5 plat : : 1 :	2.0	4.6	1.0	0.9	1.7	3.0	1.5
K_2O plat: K_2O, P_2O_5 plat : : 1 :	1.6	3.1	1.0	1.0	1.1	2.3	1.2
N, K_2O plat: N, K_2O, P_2O_5 plat : : 1 :	1.6	4.0	1.0	1.0	....	....	....

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[1] Liming this soil has rendered the phosphoric acid less deficient in spite of the fact that under ordinary conditions the deficiency would be expected to become greater the longer the experiment is continued.

It will be evident from the above table that numbers greater than 1 show an increase in yield from the use of phosphoric acid, while those equal to or less than 1 show no increase. Comparisons of the percentages of phosphoric acid dissolved by N/5 hydrochloric and nitric acids show fairly concordant results in most instances. The same chemist extracts by one acid more or less than by the other, depending upon the sample of soil. In the case of a given soil, No. 4, one chemist obtains the most phosphoric acid by extracting with hydrochloric acid, while another obtains the most with nitric acid. Mr. Peter determined the phosphoric acid dissolved by N/5 hydrochloric and nitric acids from the soils distributed by the referee for 1897, and found, respectively, 0.0039 and 0.003 in the case of one soil, and in another 0.0357 and 0.029.

From the data thus far obtained one would say that any indications of the amount of assimilable phosphoric acid which the weak hydrochloric acid solvent might furnish would be afforded equally well by the same strength of nitric acid. Therefore, if N/5 nitric acid should prove to be a valuable solvent for determining relative amounts of assimilable potash in soils there would seem to be no reason, so far as the present results indicate, why it may not replace hydrochloric acid for the determination of assimilable phosphoric acid and thus secure the unquestionable advantage of making both determinations from a single solution. It should be noticed that less phosphoric acid was extracted from No. 3 by nitric than by hydrochloric acid; according to the crop results the nitric acid afforded the best indication of the amount of assimilable phosphoric acid in this soil. The percentage of phosphoric acid from Nos. 1, 2, 3, and 4 has increased in the direction of the increase in crop production as influenced by the same ingredient. According to the analytical results the California soil, No. 5, contains no greater percentage of assimilable phosphoric acid than the most deficient soil, No. 1. This does not agree with the impression which was received concerning the relative productiveness of these two soils, but in the absence of a soil test conducted upon the California soil in a similar manner as that upon soil No. 1 the writer is not inclined to conclude that this is an instance where our provisional method for assimilable phosphoric acid has entirely failed us. Upon a previous page it was stated that farmers had after a time found it to their advantage to supply phosphoric acid to soil represented by No. 5. A soil test conducted in pots upon lots of soil truly represented by the samples under discussion would be desirable before making a definite statement regarding the relative amounts of assimilable phosphoric acid in equal weights of these soils. Certain observations to be presented in another paper lead to this opinion. If it be conceded for the time being that the crops drew their plant food from soil equal in depth to that at which the two samples, Nos. 1 and 5, were taken, viz, about 6 to 12 inches, respectively, there would be at their disposal per acre in soil No. 1, 31.8 pounds of assimilable phosphoric acid, and in soil No. 5, 51.2 pounds. These quantities are obtained by accepting 0.002 as the average percentage of phosphoric acid obtained by extracting with N/5 acid from each soil and multiplying by the weights of the soils per acre at the depths sampled, given on a preceding page. An excellent illustration is here afforded of the different conclusions which might be drawn depending upon the point of view. If the percentages only were taken into consideration it might be said that the amount of phosphoric acid at the disposal of the plants was the same in both soils, but when the weight of surface soil is also taken into account there is a great deal more phosphoric acid for the crop in soil No. 5 than in soil No. 1. According to the former way of looking at the matter our provisional method would be pronounced unreliable, but from the latter standpoint it would be said that the method gave results furnishing indications according in a general way with the crop records. It is hoped that this illustration may discourage the custom often followed of considering the percentages only of a given ingredient found by the analysis of two soils of different specific gravities, depths, and humidity. Many factors must be considered before a method which has often proved useful should be abandoned.

LIME.

Calcium oxid extracted by N/10 acetic acid.

Analyst.	Soil No. 3, deficient in calcium carbonate.	Soil No. 4, abundance of calcium carbonate.	Soil No. 5, abundance of calcium carbonate.
Thorn Smith, Moscow, Idaho	0.0209	0.1248	0.1030
Davidson and Ellett, Blacksburg, Va	.0068	....	.0906
B. L. Hartwell, Kingston, R. I	.0166	....	....

According to the above results, dilute acetic acid indicates very well the difference in the content of the more active lime compounds which exists between the Rhode Island sample and those from California.

The directions for the soil work included the description of a device for maintaining the saturation of carbonated water throughout the entire time during which the soil was to be digested. By this means fresh carbon dioxid could be readily admitted whenever the strength of the solution had been lessened by its action upon the soil, and a maximum dissolving effect be exerted. The ease in thus maintaining a constant strength gives to carbonated water an advantage not possessed by other weak solvents. It is to be regretted that no results have been reported upon the use of water kept saturated with carbon dioxid.

HUMUS.

Determination of humus in soil samples Nos. 3, 4, 5.

Analyst.	Soil No. 3.	Soil No. 4.	Soil No. 5.
	Per cent.	*Per cent.*	*Per cent.*
J. A. Bizzell, Raleigh, N. C	....	1.56	2.17
Davidson and Ellett, Blacksburg, Va	3.58	....	....
B. L. Hartwell, Kingston, R. I	4.00	....	....
Thorn Smith, Moscow, Idaho	3.99	1.59	....

The principal object in requesting determinations of humus was to see if there would be a satisfactory concordance between the results from different chemists. The results upon soil No. 3 show that some difficulty exists in obtaining parallel results under varying circumstances. Davidson writes: "The per cent of humus found will depend very largely on the amount of extraction with 1 per cent hydrochloric acid solution. The more you extract the less humus you get. As an experiment we ran one determination using about 500 cc of 1 per cent hydrochloric acid, and the per cent of humus from this was 3.14." The writer also found that by continuing the extraction with hydrochloric acid for some time after there ceased to be a reaction for lime, varying results were obtained, depending upon the amount of the extraction with hydrochloric acid, viz, 3.28 per cent, 2.88 per cent, and 2.77 per cent. The result published in the table above was an average of 3.98 and 4.01 per cent, which were obtained by running two tests side by side and stopping the extraction with hydrochloric acid as soon as the lime was removed. Emery[1] and Rimbach,[2] in their articles on humus, point out the fact that after the ammonia extract is evaporated and dried at 100° a certain amount of ammonia is retained by the residue which, according to the official method, would pass off upon ignition and cause a plus error. Rimbach ascertained the extent of this error by previously boiling another aliquot part of the ammonia extract to expel free ammonia and then distilling the residue with magnesia. The quantity found by the latter process was subtracted from the loss of weight by ignition in obtaining the true humus. Rimbach also concludes that by leaching with hydrochloric acid and water a certain quantity of humus, varying in the different soils, is lost to the subsequent extraction with ammonia. Snyder[3] calls attention to the fact that certain soils give up some of the phosphoric acid to the dilute hydrochloric acid which otherwise would have been included in the ammonia extract. The above considerations suggest the possibility that the long extractions with hydrochloric acid may have removed from soil No. 3 certain ingredients which otherwise would have been extracted by the ammonia, and thus have increased the amount of so-called humus either directly or by virtue of having retained ammonia which could not be driven out at 100°.

ASPARTIC ACID METHOD.

This method, which was brought before the association in 1898 by Mr. Maxwell, of the Hawaiian Experiment Station,[4] consists essentially in digesting the soil twenty-four hours with a 1 per cent aspartic acid solution, shaking gently every fifteen minutes during the daytime. At the request of the referee Mr. Maxwell expressed a willingness to test the soils which have been under consideration. Unfortunately only one of the samples, the arid soil from California, No. 4, was analyzed. The following percentages were reported: Calcium oxid, 0.1334; potassium oxid, 0.0170; phosphoric acid, 0.0294. For comparison with these amounts the average results from 30 Hawaiian soils were also reported, as follows: Calcium oxid, 0.1400; potassium oxid, 0.0406; phosphoric acid, 0.0025. The following is quoted from Mr. Maxwell's letter of June, 1899: "These results justify the warning statement made by me a year ago, viz, 'The results obtained with Hawaiian soils which are ultra basic in composition must not be accepted as a guide in judging of the state of availability of the elements in old siliceous soils.'" Our provisional method for assimilable phosphoric acid gave more than four times the amount of phosphoric acid obtained by the aspartic acid method, while the amounts of potassium and calcium oxids extracted by the aspartic acid are very similar to those obtained by the weak solvents which have been used by this association.

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[1] Jour. Am. Chem. Soc., 22,285.

[2] Jour. Am. Chem. Soc., 22,695.

[3] Jour. Am. Chem. Soc., 16,210.

[4] A. O. A. C. Proceedings, 1898, p. 63.

REMARKS AND RECOMMENDATIONS.

A study of the work of this association in connection with the determination of assimilable plant food impresses one with the necessity of publishing all of the available data concerning the soils employed which may assist in an interpretation of the analytical results. A simple statement from a referee of the comparative deficiency of a certain ingredient in various soils is not sufficient when this statement may mean the acceptance or rejection of a method which has been tried. The facts upon which the referee bases his conclusions should be either published in his report or reference made to some other published work in which they occur. The amount of assimilable plant food in soils is so liable to fluctuation that even the most complete data obtainable is very apt to prove inadequate.

The wide differences in the results from different chemists working upon the same sample of soil render comparisons of results upon two soils by different analysts practically useless when the content of the soils does not vary widely. The work of the present year would have been much more valuable had the chemists, when undertaking the determination of a given ingredient, not omitted some of the soils upon which the determination was desired. The results of a given chemist working under definite conditions upon a number of soils may be profitably compared with one another, but a glance at the results contained in this report will show that such comparisons were possible in very few cases only. Your referee can do no more than present as a report of progress such data as have been obtained concerning the recommendations received, since the analytical results do not justify drawing any conclusions at this time. The recommendations made to the present referee must remain, therefore, as unfinished business to be carried on by my successor unless he be relieved by the association.

It is recommended that determinations of phosphoric acid be made by N/5 nitric acid as well as by N/5 hydrochloric acid, for reasons which have been mentioned previously.

The recommendation made last year, that a 3-mm sieve be adopted where the soil is to be used for determinations requiring 100 grams or more, is hereby repeated.

The referee's report should contain such data as are necessary for converting the percentage of an ingredient to the number of pounds of the same in a given area of soil at the depth sampled.

At this stage of the proceedings Dr. William McMurtrie, of New York, ascended the platform, at the invitation of the president and amid the hearty applause of the members of the association.

The PRESIDENT. I see the association recognizes an old friend, and it is with very great pleasure that I introduce to the members of the association Dr. McMurtrie, the president of the American Chemical Society.

Dr. McMURTRIE. It is with genuine pleasure, I assure you, that I receive such a greeting from this body to the organization that I have the honor to represent here in an executive capacity. It illustrates very thoroughly to me the interest that you have in the organization of American chemists, and I can well understand the sympathy that you have for it, in that you are surely all members of that organization; if you are not you should be. I can sympathize as warmly with you in the work that you are doing. I was brought up in it, and all of us know how difficult it is to forget the first love.

This discussion that you have just listened to has been particularly interesting to me, because it concerns a kind of work that I had to do in my early experience in connection with agricultural chemistry, and there is nothing, I am sure, that will do as much to advance the agricultural interests of the country. In this statement there is nothing derogatory to the other lines of work which you are doing, and you are to be congratulated upon the opportunities that are afforded you by the enormous success that you have attained in your work in the experiment stations and in the other organizations that you represent. I sympathize fully with you in your work, because I had something to do with the efforts that led up to the establishment of those stations, and I was proud to be associated in that work with such men as Cook, Appleton, Goessmann, and the other men who worked so hard for the passage of the Hatch bill, which brought about the organization of these stations devoted to agricultural science. And we all have a right to be proud of the success that has been attained. We all know how common it was formerly for those wishing further instruction in agriculture to go across the water and seek assistance from our German friends, and surely no one could fail to appreciate and draw inspiration from the work of the leader of agricultural chemistry, the great Liebig. But times have changed and we find that the inspiration and influence are going the other way. We find that our friends across the water come to us for instruction, and we may feel proud that the results of our work have been such that we can confidently point out to them some of the lines in which it will be possible to secure the further advancement of our science.

The changes in the work of chemistry in this country and the great advances that have been made are confined not alone to what has been done in agriculture. It has been interesting to me to follow the work of a young friend who has been abroad for the past two years to complete his education in metallurgical lines. He went to one of the great institutions of Germany, and said that he was greatly disappointed to find that the year's work he was called upon to do was only a repetition of his last year's work in this country. He went to the University of Berlin and attended a course of lectures, and was well satisfied, but when he desired to take up the micrographic study of iron and steel for his doctor's degree and looked around for the material for that work, he found it impossible to obtain the apparatus for the work outside of the Reichsanstalt in Charlottenburg. A few weeks later he met President Low, who said that Columbia University was very well fitted for that kind of work, and he decided to come back to the United States to complete the work for his doctor's degree. It seems to me that these things are always of the greatest encouragement to the chemists of the country.

We have further encouragement, not only in what is being done in research work, but also in applied chemistry. I suppose that in no age of the world, nor in any part of the world, has the progress in industrial chemistry been so great as in this country during the past decade. We find that new industries are springing up all the time; we find that the demand for our products increases at home and increases abroad, and that there is a most gratifying growth in the exports of chemical manufactures. It will interest you, I am sure, to study the statistics of exports of our manufactures of chemical products and note the enormous increase in the last decade. It runs into the millions of dollars, and even into the hundreds of millions of dollars.

Another encouraging thing to me with regard to the growth and progress of chemistry in this country is the demand for expert and accurate work in technology. You are the representatives of probably the most important branch of chemical technology. You know what is necessary to progress; you know what is necessary to the obtaining of good results. You know that all the accuracy of manipulation, all the depths of thought of the careful investigations that belong to any research laboratory, belong to your laboratories; and what you are doing in your laboratories is being done largely in this country in the laboratories of all the leading and successful manufacturers. It is becoming a rare thing nowadays to find an important manufacturing establishment without its research laboratory; and it is interesting to note that what I had occasion to say a few years ago to one of my friends, who was greatly devoted to research work in chemistry and was inclined to sneer at technical work, has been thoroughly borne out. In talking with this young man one day I remarked, "You have had occasion to make some careful determinations of atomic weights; how many determinations do you think it would be necessary for you to make before you would be willing to accept the results?" "Oh, from six to ten," he answered. "Well," I said, "if you were working in my laboratory and couldn't make two determinations and have them agree we wouldn't want you."

The young man's opinion of the work required by the industries has been very materially changed. He has some appreciation of the fact that when a business man asks a question he expects to have that question answered promptly and accurately. It is such questions that come to you, and which you are answering every day, and you may be proud to know that the country feels that you are answering these questions promptly and that you are doing it accurately.

The President. It is impossible for any one man to cover the broad field of chemistry. It is therefore a special pleasure for us to have men in other branches of work, and those who have felt interest in our line of work, look in upon our proceedings and talk to us, and I thank

Dr. McMurtrie in the name of the association for the trouble which he has taken this morning.

Mr. HARTWELL. I have a paper which I would like to present.

The PRESIDENT. We will hear the paper.

A POT EXPERIMENT TO TEST FIELD OBSERVATIONS CONCERNING SOIL DEFICIENCIES.

By BURT L. HARTWELL.[1]

Certain impressions which resulted from a study of the results secured by this association over a series of years in connection with soil work were outlined at the San Francisco meeting in July, 1899, in my report[2] as referee on soil analysis. As the investigation presented in the present paper was undertaken as a result of previous consideration of the points presented at that time, brief mention will be again made of them here. Much analytical work has been done by the association upon samples taken from differently fertilized plots of the same soil. The existence of such plots resulted from a quite extended application of the method of determining the needs of soils by a soil test conducted in the field. Determinations of the total potash, for example, have been made in samples taken from plats which had received no applications of potash and also from those to which potash had been abundantly supplied. In a number of cases the records were sufficiently complete to enable one to calculate approximately the difference to be expected in the percentages of potash in soils from plats receiving potash and from those not receiving it. In making such calculations the applications of potash and the amount of the same removed by the crops during the series of years in which the experiments had been conducted were considered. No data were available for determining the relative loss by drainage, so that the influence of this factor could not be determined. Some of the soils in question were deficient in natural drainage, and it does not seem probable, therefore, that the loss by drainage would be of sufficient importance to explain certain of the differences to be noticed. The figures given below represent the differences obtained by subtracting the percentages of potash found in soil of the no-potash plats from those obtained in soil to which potash had been added. For comparison with these are given also the differences in the net amounts of potash added, found by calculation in the manner referred to above.

Differences between the percentages of potash in the no-potash and potash plats.

Method.	Kentucky soil.	Rhode Island soil.	Indiana soil.	Indiana soil.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Found by J. Lawrence Smith method	.018	–.063	–.081	.078
Found by official method (HCl. 1.115 sp. gr.)	.015	.004	.021	.022
Found by calculation	.043	.046	.008	.018

NOTE.—The results in the case of the official method were deduced from the average of a number of determinations by different chemists. Those by the J. Lawrence Smith method were from the work of single chemists.

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[1] H. J. Wheeler rendered valuable assistance by his advice and interest in the work. A. W. Bosworth attended to the drying and weighing of the crops.

[2] Bul. 57, Div. of Chem., U. S. Dept. of Agr., p. 74.

It may be seen from the above results that there is not the conformity which one would expect between the analytical results and those obtained by calculation. In every case, considering the history of the plats, a plus difference should be expected, as shown by the last row of figures, and yet the total amounts of potash as found by the Smith method are, in two of the four cases, less from the plats receiving potash than from those which did not receive it. The official method does not give the total amount of potash, and the results secured should not be expected to conform to the calculated results as nearly as those by the Smith method. The analytical data here presented do not, as a whole, furnish reliable information as to the excess of potash in one plat over another as a result of increased fertilization with this element. Similar observations were made regarding phosphoric acid. The writer is not inclined to question seriously analytical results representing the average determinations of a number of chemists working with a given sample, although there may be a lack of confidence where the results are those of a single chemist, but in view of the difficulties in obtaining a sample which truly represents a plat of land it seems more probable that in all cases proper care is not exercised in the collection of samples. Failure to appreciate the care necessary in this connection may render valueless the careful work of a large number of chemists.

This association, in its attempts to find solvents which would extract from different soils amounts of potash and phosphoric acid bearing relations similar to the crop-producing power, has compared the analytical results obtained from soils claimed to be deficient in an ingredient with those obtained from soils of high fertility, and consequently containing an abundance of all necessary ingredients. If the solvent failed to extract amounts agreeing with the claimed productiveness of the soils, it was finally abandoned as unsuited for the desired purpose. It becomes evident, therefore, that no doubt should exist as to the amount of the element in question, which is really in a suitable form for the production of plants, otherwise a useful solvent may be discarded because of an incorrect knowledge of the soils upon which it is tried. The soil's natural productiveness as influenced by the particular ingredient under investigation should be known with certainty before any conclusions regarding a solvent or method should be indulged in. The only reliable standard in this work is the crop which a given soil ingredient can produce when the other ingredients of plant food are present in proper conditions and amounts, and when all other factors aside from plant food are favorable. It will be readily seen that such conditions, even though they may be plentiful, are not easily recognized with certainty. It is obvious that a highly productive soil can not be deficient in any necessary element, for the product of a soil as influenced by the plant food contained in the same depends upon the amount of the most deficient ingredient. It does not follow, however, that a less productive soil contains less available plant food, for unfavorable conditions which escape the observer's notice may be the cause of the decreased yield. It must not be concluded that because one soil furnishes 10 bushels of wheat more than another that a similar difference should necessarily appear in the amount of potash or phosphoric acid dissolved by a given solvent. A decidedly unproductive soil may be well supplied with available nitrogen, phosphoric acid, and potash. It is, then, no easy matter to know with certainty the assimilability of a particular soil ingredient, and yet without such assurance it is impossible to judge of the merits of a given solvent for the purpose under discussion. No doubt many incorrect conclusions have been drawn because sufficient attention was not given to finding out the true nature of the soil which was taken as a standard by which to measure the value of a particular solvent.

Perhaps a well-conducted soil test is the most satisfactory method of arriving at a knowledge of the particular deficiencies of a worn-out soil. Trustworthy information can not be obtained, however, in regard to the availability of the nitrogen, phosphoric acid, and potash in a soil under experimentation unless precautions are taken that all other conditions are favorable to plant growth. A test conducted upon acid soil may, for example, lead to very different conclusions from those which would have been drawn had lime been applied previously in sufficient amount to render the reaction of the soil suitable for the best growth of the crop used in the experiment.[1] There are indications that in some localities magnesia may be lacking in the soil, in which case a soil test should not be conducted without the addition of this constituent.

As soon as the writer received his appointment as referee in the fall of 1898 there was sent to the station chemists a circular letter, published in our last proceedings, which contained a request for soils believed to be deficient in some particular ingredient of plant food. As a result of this inquiry soils were received from Massachusetts and Indiana said to be deficient in assimilable phosphoric acid and two from Connecticut supposed to be lacking assimilable potash. All of the lots were collected in the spring of 1899.

The object in securing these soils was to test them in pots, with special care to supply in the fertilization ingredients other than nitrogen, phosphoric acid, and potash which they might possibly need and which are often overlooked in soil tests. It seemed to the writer, in view of the considerations which have been presented in this paper, that such a course was necessary in order that the assimilability of a given ingredient might be determined under well-controlled conditions and that the samples used for analysis might be taken from the thoroughly mixed soil and leave no doubt about their being representative.

Before describing the pot experiment such records of the soils as it has been possible to secure will be given.

FIELD RECORDS.

Soil from Wapping, Conn., farm of H. W. Sadd.[2]—Sample taken to the depth of 6 inches from seven different points on an acre—a light loam, fine in texture, with a porous subsoil. The field from which this soil was taken, previous to 1891, was an old pasture that had not been plowed or fertilized for years. In 1891 a soil test with indian corn was conducted upon this field under the direction of the Agricultural Experiment Station, Storrs, Conn., resulting in the following yields per acre.[3]

Yield per acre of indian corn raised in soil test at Wapping, Conn., 1891.

Plat.	Fertilizers supplied.	Shelled corn.	Stover.
		Bushels.	*Pounds.*
0 00 Av.	None	31.1	2,305
A	N	29.6	2,170
B	P_2O_5	30.0	2,130
C	K_2O	42.6	2,900
D	N, P_2O_5	37.5	2,680
E	N, K_2O	49.4	3,470
F	P_2O_5, K_2O	40.5	3,510
G	N, P_2O_5, K_2O	49.8	4,680
H	Plaster	32.1	2,230

A crop of corn and one of grass had been grown upon the field and an application of stable manure made since the field test was conducted. In taking the sample the manure which then remained on the surface was avoided as much as possible.

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[1] See Bul. 46, R. I. Agr. Expt. Sta., p. 97.

[2] Location about lat. 41° 50′ and long. 72° 33′, on the soil map (Hartford sheet) of the Div. of Soils, U. S. Dept. Agr., and belonging thereby to the Triassic stony loam.

[3] Storrs Agr. Expt. Sta. Report 1891, p. 192.

Soil from Limerock, Conn., farm of M. H. Dean.—"A striking peculiarity of the soils of this region (the Housatonic Valley) is the presence of large quantities of phosphates. The soil is alluvial, and was apparently formed from the decomposition of limestone formations located along the Housatonic Valley to the northward. This limestone evidently contains phosphates, and by its decomposition these phosphates must have been added to the soil."[1]

Soil tests[2] have been conducted under the direction of the Agricultural Experiment Station, Storrs, Conn., upon the field from which this soil was taken. Below are given the yields per acre:

Yield per acre of maize and rye raised in soil tests at Limerock, Conn., 1889–1893.

Plat.	Fertilizers supplied. *a*	1889, maize.		1890, maize.		1891.	1892.	1893, maize.	
		Shelled corn.	Stover.	Shelled corn.	Stover.		Unthrashed winter rye. *b*	Shelled corn.	Stover.
		Bu.	*Lbs.*	*Bu.*	*Lbs.*		*Lbs.*	*Bu.*	*Lbs.*
0	None	9.9	1,470	3.9	1,320	Cowpeas; plowed under; seeded to rye.	3,130	10.3	2,230
A	N	16.9	1,890	12.9	1,990		3,740	16.2	2,580
B	P_2O_5	9.1	1,670	5.0	1,470		3,840	8.9	2,260
C	K_2O	18.8	3,340	7.2	4,390		4,400	28.5	4,310
D	N, P_2O_5	18.0	2,770	17.0	2,300		4,180	22.0	3,340
00	None	14.7	1,790	7.3	1,590		4,060	19.3	2,550
E	N, K_2O	25.3	2,890	44.4	4,630		4,600	32.6	3,830
F	P_2O_5, K_2O	17.4	2,950	8.8	4,300		4,470	36.9	5,250
G	N, P_2O_5, K_2O	22.1	3,050	35.0	5,850		4,610	39.5	5,400
Ga	N (double amount), P_2O_5, K_2O.	....	....	40.4	5,860		4,470	42.8	5,800

a The N, P_2O_5, and K_2O were furnished, respectively, by nitrate of soda, dissolved boneblack, and muriate of potash.

b Red clover followed the rye.

Previous to the time of the above tests the soil had been apparently exhausted by cropping without manure. Since 1893 the following crops have been grown without fertilizer, viz: 1894, oats; 1895, rye; 1897, buckwheat, rye; 1898, maize. In 1896 potatoes were grown and one-half ton of a complete fertilizer was applied.

Soil from Amherst, Mass., furnished by the Massachusetts Agricultural Experiment Station from Plat 0, Field F;[3] a friable loam, with a retentive subsoil of clay that is nearly filled with gravel and small stones in the lower strata. Previous to 1887 the field had been used as a meadow and was well worn-out, at that time yielding but a scanty crop of English hay.

The plat (No. 5) in proximity to the one from which the sample was taken and with which comparisons will be made later received the following treatment: In 1887 the sod was turned under. The crops grown in 1888 and successively have been Hungarian grass, forage crops, potatoes, wheat, serradella, maize, barley, rye, soy beans, Swedish turnips, and maize. The fertilization began in 1890 with about 500 pounds of dissolved boneblack, 290 pounds of nitrate of soda, and 390 pounds of potash-magnesium sulphate. This application was made yearly till 1895, after which time no more phosphoric acid was added, but the yearly addition of the other ingredients was increased one-half. In 1893 and thereafter the same crops were grown upon the plat from which the sample of soil was secured (No. 0) as upon the above-mentioned one, but the first addition of fertilizer was made in 1896, since when the application of nitrogen and potash has corresponded with the other plat. So far as is known, no phosphate has ever been added to plat 0. Both plats were limed at the rate of 1 ton of quicklime per acre in the spring of 1898. It may be seen that during 1896, 1897, and 1898 the fertilization of the two plats was the same, but previous to these years plat 0 had received no fertilizer while plat 5, during a series of years, had received a total application of about 1,900 pounds of nitrate of soda per acre, 2,500 pounds of potash-magnesium sulphate, and 2,500 pounds of dissolved boneblack. With the differences in the treatment of the two plats in mind let us compare the yields in pounds per one-tenth acre plat:

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[1] Storrs Agr. Expt. Sta. Rept., 1893, p. 123.

[2] Storrs Agr. Expt. Sta. Rept., 1889, 1890, 1893.

[3] Hatch Expt. Station, Mass., 10th An. Report.

Yield in pounds per one-tenth acre plat, Amherst, Mass., soil, 1896–1899.

Plat.	1893, maize.		1894, barley.		1895, rye.	1896, soy bean.		1897, turnips.	1898, maize.	1899, oats.	
	Ear corn.	Stover.	Grain.	Straw.	Entire crop.	Beans.	Straw.	Roots.	Stover.[a]	Grain.	Straw.
	Lbs.	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*
Plat 0 (N, K_2O)	0	366	3.4	28	269	64	182	467	267	107	203
Plat 5 (N, K_2O, P_2O_5)	349	502	76	217	402	159	318	1,041	457	134	260

[a] Owing to damage by a violent storm, the stover furnished the best comparison.

I am informed that cabbages were upon the plats in 1900, and that there was almost nothing on plat 0, while upon plat 5 there was a very good crop.

Soil from Orleans, Ind., farm of Frank Turley.—A medium clay resting on dark red clay, which in turn rests on the limestone rock of the region. The combined thickness of the two soils is from 2 to 6 feet. The land has been under cultivation for about seventy-five years, and at one time was so depleted that in an ordinary season it would produce without fertilization only 5 to 8 bushels of wheat. One hundred pounds of ground bone per acre, a quite common application upon this land, increased the productiveness to 15 bushels. A yield of 30 bushels of corn in a favorable season on an unfertilized plat was increased to 38 bushels by an application of acid phosphate and potash. An addition of nitrate of soda gave no further increase, nor did increasing the amount of potash from 30 to 60 pounds per acre. The "Turley" soil upon which some analytical work was done by Huston and Bartlett[1] was taken from the same location as the soil under experiment.

RECORD OF THE POT EXPERIMENT.

As a guide in watering the pots approximate determinations of the per cent of water which the soils were capable of absorbing were made. One hundred parts of the water-free soil were found capable of absorbing the following parts of water, viz: Wapping soil, 63 parts; Limerock soil, 45; Amherst soil, 47; Orleans soil, 46.

All of the soils when tested with blue litmus paper imparted a red color to the same. The Wapping soil, however, gave a more marked change than the others. The humus dissolved by treating a soil directly with ammonia water without previous extraction with hydrochloric acid is sometimes called free humic acid, and is taken as a measure of the need for liming. Qualitative tests with ammonia water were made by noticing the intensity of the color of the extracts. The extract from the Wapping soil was considerably darker than that from the Limerock and Amherst soils. The Orleans soil yielded no color to the ammonia water.

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[1] A. O. A. C. Proceedings, 1896, p. 91.

Six Wagner pots, about 8 inches in diameter by 8 inches high, were used for each of the four soils. An amount of the natural soil equivalent to the following weight of completely dried soil was used per pot in each case, viz: Wapping soil, 13 pounds; Limerock soil, 14.2 pounds; Amherst soil, 16.3 pounds; Orleans soil, 13 pounds. In the case of each soil two of the six pots were left without any addition of fertilizer and two received the following:[1]

	Grams.
Nitrate of soda	1.0
Dried blood (15.41 per cent N in dry matter)	2.5
Dissolved boneblack	6.0
Muriate of potash	.8
H. G. sulphate of potash	.8
Calcium carbonate, c. p	10.0
Hydrous magnesium sulphate	1.5

In the case of the Wapping and of the Limerock soils (supposed to be deficient in potash) two of the six pots received all of the above-mentioned materials, except that the potash salts were omitted, and in the case of the Amherst and of the Orleans soils (supposed to be deficient in phosphoric acid) two pots received all of the materials except the dissolved boneblack. This gives, then, in each instance two pots with only the soil as received, two with complete fertilization, and two with complete fertilization except that the supposed deficient ingredient is omitted.

In June, 1899, 50 selected kernels of oats were planted in each pot. When the plants were well up they were thinned to 23 in each pot. Whenever the plants of a particular group required it, sufficient water was added to the pots so that the soil should be about 70 per cent saturated. The weights of the crop at maturity will be given later in this paper in connection with those of 1900.

In the spring of 1900 the soil was emptied from each pot and, after breaking the lumps, replaced. Oats were planted as in the previous year, but the plants were thinned to 35 instead of 23. The only fertilizing ingredient added was 1.5 grams of c. p. ammonium nitrate to each of the 24 pots. This was added in solution at the time of the first watering and was intended to serve as a ready source of nitrogen for the young plants in case the conditions had been unfavorable to the nitric ferment in the dry soil of the pots during the winter season. The crop was harvested at maturity during the first week in August and barley planted in its place. The barley had just begun to head in the most forward pots when it became necessary to harvest it. The weights are not included in this connection because the crop had not had time to dry, and reference to this crop will be made only incidentally when the results bear particularly upon the discussion of those obtained with oats. (See Pl. I, fig. 1.)

Yields (in grams) from the pots containing the Wapping soil.

Materials supplied. (See above.)	Pot No.	1899, oats.				1900, oats.[a]			
		Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).	Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).
None	1	8.25	8.93	3.04	5.89	20.58	23.63	8.63	15.00
	2	7.68	8.28	2.24	6.04	28.39	31.79	11.78	20.01
All[b] except potash	3	24.00	25.86	9.77	16.09	36.46	41.40	13.01	28.39
	4	23.51	25.37	8.12	17.25	35.32	39.71	11.14	28.57
All[b]	5	22.31	24.06	7.69	16.37	45.61	51.89	14.34	37.55
	6	22.55	24.39	7.42	16.97	40.73	45.48	16.92	28.56

[a] All pots received 1½ grams ammonium nitrate.

[b] Including nitrogen, phosphoric acid, potash, lime, and magnesia.

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[1] Multiplying these numbers by 293 gives the number of pounds per acre.

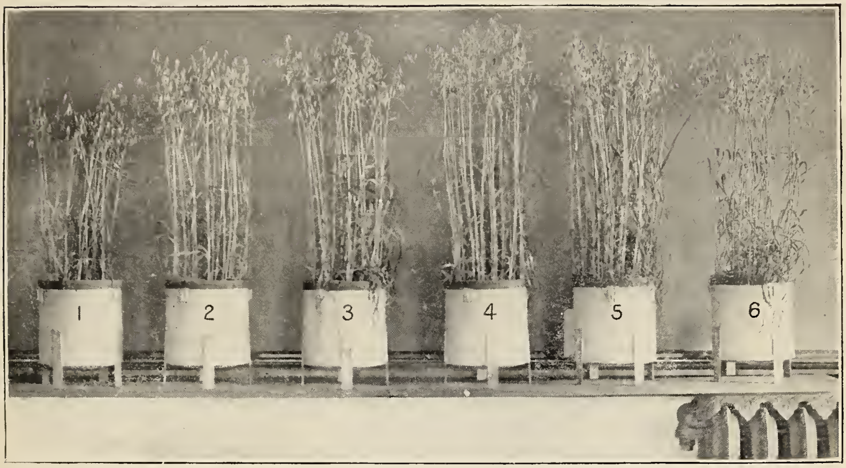

FIG. 1.—OATS GROWN IN WAPPING, CONN., SOIL, 1900.

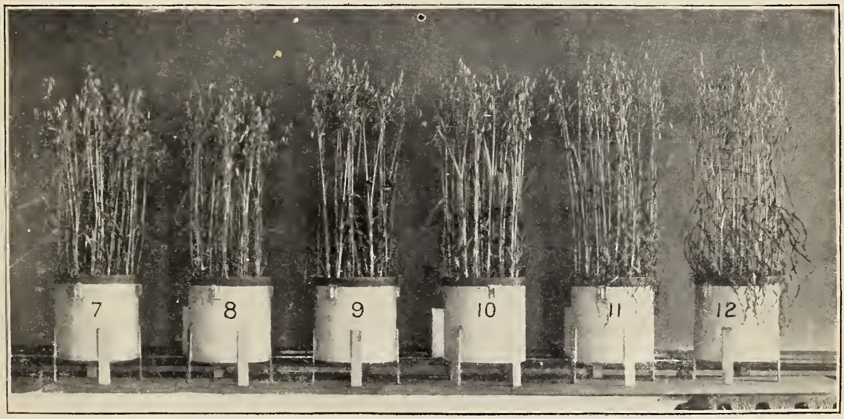

FIG. 2.—OATS GROWN IN LIMEROCK, CONN., SOIL, 1900.

By reference to the field test with maize on this soil in 1891 (page 75) it may be seen that the results indicated undoubtedly that potash was the ingredient chiefly lacking at that time. The addition of nitrogen with the other ingredients also increased the yield in each case, although to a considerably less extent than the potash did. Phosphoric acid was evidently not needed.

The results of the pot experiment, however, indicate that under the conditions of the experiment no potash was needed the first year for the production of a normal crop of oats when the other ingredients were supplied; in fact, the yield was somewhat lessened where the potash was added. The second year's results, however, showed some gain from the use of potash. In 1899 the yield from the pots (Nos. 1 and 2) containing the natural soil was vastly less than from those receiving everything except potash (Nos. 3 and 4), but in 1900 the relation was much closer. It should be recalled that 1½ grams of ammonium nitrate were added to each pot in 1900; this fact may explain the relative increase upon pots 1 and 2 which had previously received nothing. Considering that nitrogen was somewhat lacking in this soil in 1891, even though sod, which must have supplied this ingredient to some extent, was turned under previous to the field test, it is probable that the lack of available nitrogen in the soil as received was greater than that of potash. It is evident, considering the preliminary tests with litmus paper and with ammonium hydroxid, that the application of some basic ingredient, such as air-slaked lime, is of first importance for the best growth of most crops upon this soil; this is indicated by the fact that the barley, which is more susceptible than oats to acid conditions, failed almost completely in pots 1 and 2.

The only object in view in planning the pot tests with the various soils was to determine whether the supposed greatest deficiency really existed under the conditions of the experiment and to gain some idea of its extent. The number of pots was therefore limited and was insufficient for drawing conclusions regarding other ingredients. The results from three of the four soils have been so surprising that it has been necessary to look for other factors which evidently exerted more influence than those which were expected to control the crop results.

Yields (in grams) from the pots containing the Limerock soil.

Materials supplied. (See page 78.)	Pot No.	1899, oats.				1900, oats.[1]			
		Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).	Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).
None	7	12.19	13.23	5.37	7.86	26.78	30.20	11.01	19.19
	8	11.61	12.66	4.92	7.74	25.59	28.61	11.03	17.58
All except potash[2]	9	24.89	26.99	9.29	17.70	30.43	34.17	10.59	23.58
	10	24.53	26.49	7.84	18.65	30.88	34.63	11.27	23.36
All[2]	11	24.44	26.40	8.54	17.86	31.36	35.35	12.31	23.04
	12	22.77	24.71	7.96	16.75	35.15	39.63	14.14	25.49

[1] All pots received 1½ grams ammonium nitrate.

[2] Including nitrogen, phosphoric acid, potash, lime, and magnesia.

According to the field tests conducted upon the above soil from 1889 to 1893 (see p. 76), potash was the constituent chiefly deficient. Nitrogen proved of some value, however, even the first year after the original sod had been turned under; the second year its benefit in the production of shelled corn was still more marked, even exceeding that of potash. After cowpeas had been plowed under in 1891 and nitrogen thus supplied, the chief deficiency during the remainder of the experiment was undoubtedly potash. The addition of dissolved boneblack in the earlier years of the experiment seemed detrimental, and there is little evidence at any time that phosphoric acid was deficient. (See Pl. I, fig. 2.)

The above results in the pots are very similar to those with the Wapping soil in showing a slight decrease from the use of potash the first year, but some increase the second year. It may be noticed also that the marked difference in the first year's crop between the yield from the natural soil (pots 7 and 8) and that to which all the ingredients except potash were added (pots 9 and 10) largely disappears after the addition of ammonium nitrate preparatory to the second crop of oats. Considering the results in the field, it seems probable that nitrogen in an available form may have been more deficient than potash when the soil was received. The preliminary tests with blue litmus and ammonia water and the fair growth of barley in the pots to which no calcium carbonate was added indicate much less necessity for liming than in the case of the Wapping soil.

Yields (in grams) from the pots containing the Amherst soil.

Materials supplied. (See page 97.)	Pot No.	1899, oats.				1900, oats.[1]			
		Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).	Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).
None	13	14.20	15.27	6.70	8.57	30.11	33.65	14.65	19.00
	14	9.83	10.55	3.94	6.61	36.43	40.83	16.96	23.87
All except phosphoric acid.[2]	15	27.27	29.62	13.70	15.92	37.21	41.69	17.65	24.04
	16	26.97	29.42	13.22	16.20	39.17	43.35	18.51	24.84
All[2]	17	27.43	29.82	11.70	18.12	40.33	44.51	17.11	27.40
	18	24.81	27.12	8.81	18.31	39.32	43.47	16.45	27.02

[1] All pots received 1½ grams ammonium nitrate.

[2] Including nitrogen, phosphoric acid, potash, lime, and magnesia.

The field results (see page 77) indicate a lack of assimilable phosphoric acid in the plat from which the above soil was obtained, but it may be seen from an inspection of the yields obtained by pot experimentation that no appreciable gain resulted from the application of phosphoric acid (pots 17 and 18) in addition to the other fertilizing ingredients (pots 15 and 16); in fact, the yield of grain during each of the two years was somewhat greater where the phosphoric acid was omitted. The growth of barley was apparently no greater in the phosphoric-acid pots. (See Pl. II, fig. 1.)

The yields of oats in pots 13 and 14 compared with those from the other pots were much greater in 1900 after the addition of the ammonium nitrate than in 1899 when no nitrogen had been applied. It seems probable from this that the soil as received may have been somewhat in need of readily assimilable nitrogen. To be sure nitrate of soda had been applied in the field the spring before the sample was taken, but it is not improbable, considering the readiness with which this compound leaches and its liability to change into less available forms, that an application of readily assimilable nitrogen to pots 13 and 14 in 1899 would have been very beneficial to the crop.

In 1899 oats were also grown upon the field from which the Amherst soil was taken, not, however, until an application of nitrate of soda and potash magnesium sulphate (plat 0) had been made. It is interesting in this connection to compare once more the yield from this plat with that from plat 5, which received dissolved boneblack in addition. The yield per one-tenth acre from plat 0 was 107 pounds of grain and 203 pounds of straw; that from plat 5 was 134 pounds of grain and 260 pounds of straw. Considering the fertilization one might have expected a relation similar to this between the yield from pots 15 and 16 and that from pots 17 and 18, but the above table shows that there was no gain in total crop from the use of phosphoric acid. The application in the pot experiment of calcium carbonate at the rate

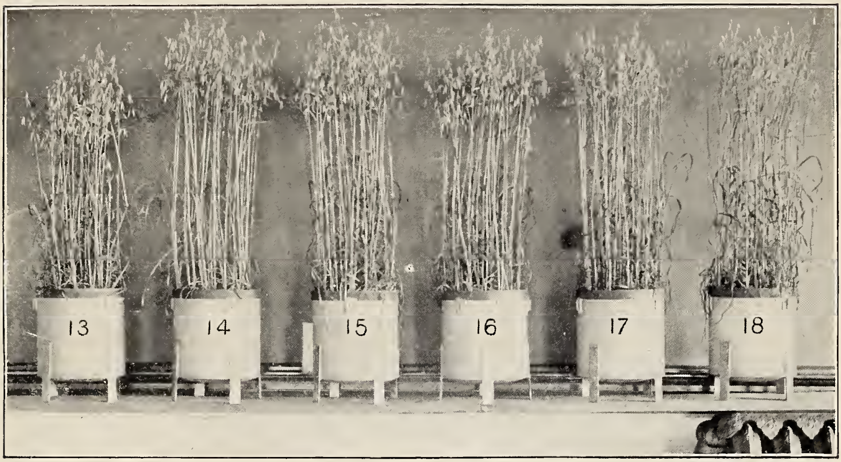

FIG. 1.—OATS GROWN IN AMHERST, MASS., SOIL, 1900.

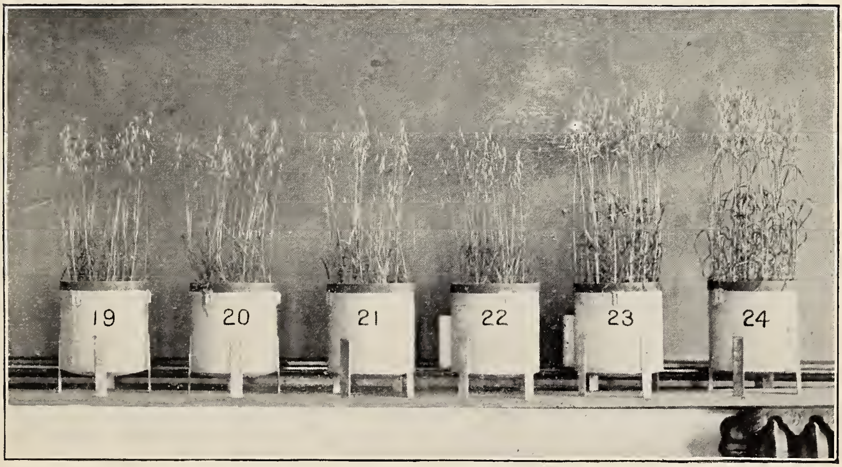

FIG. 2.—OATS GROWN IN ORLEANS, IND., SOIL, 1900.

of about 1½ tons per acre may have increased the assimilability of the phosphoric acid to such an extent that no further application was necessary. To be sure a field application equivalent to 1 ton of quicklime had been made in 1898, but considering the character of the soil it seems probable that the further application in the pots may have changed the reaction of the soil so that the conditions were more favorable than in the field to the liberation of phosphoric acid. The above view is strengthened by the fact that upon certain plats at the Rhode Island Agricultural Experiment Station which had received only nitrogen and potash, and upon which phosphoric acid was lacking, an application of 1 ton of air-slaked lime per acre rendered the phosphoric acid more assimilable but not to the same extent as 2½ tons.

Yields (in grams) from the pots containing the Orleans soil.

Materials supplied. (See page 78.)	Pot No.	1899, oats.				1900, oats.[1]			
		Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).	Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).
None	19	6.96	7.52	3.31	4.21	10.57	12.15	5.09	7.06
	20	6.53	7.11	2.86	4.25	11.11	12.72	4.93	7.79
All[2] except phosphoric acid	21	8.05	8.74	4.22	4.52	10.97	12.39	4.88	7.51
	22	9.02	9.86	4.04	5.82	9.24	10.55	4.44	6.11
All[2]	23	23.38	25.31	9.56	15.75	28.82	32.44	14.18	18.26
	24	24.19	26.25	10.26	15.99	28.88	32.24	13.33	18.91

[1] All pots received 1½ grams ammonium nitrate.

[2] Including nitrogen, phosphoric acid, potash, lime, and magnesia.

From the field observations upon the soil heretofore given it was found that nitrate of soda gave no increase, but that an application of phosphoric acid resulted in a greatly increased yield.

The above results in the pots confirm without doubt the field records concerning the lack of assimilable phosphoric acid, for the entire crop from the pots (Nos. 23 and 24) receiving all the ingredients was nearly three times as great as from those which were treated the same, except that phosphoric acid was omitted (pots 21 and 22). The lack of phosphoric acid is so marked in this soil that it seems to be about the only addition necessary for the production of a maximum crop of oats. (See Pl. II, fig. 2.)

The Indiana soil is then the only one of the four upon which the results of the two years' experiment in the pots with oats agreed fully with the field observations. Many factors may have had an influence in causing the apparent disagreement between the field and pot experiments. In the pot experiment the soil was thoroughly mixed and all large lumps broken; water was supplied whenever needed by the plants; temperature conditions were different in the exposed pots than in the earth; the crop was different in some cases from those used in the field. It can not be denied that these factors may have exerted an influence, but in view of the fact that soil tests conducted in pots have in so many instances borne out the conclusions derived by field experiments similarly conducted at the same time it seems more likely that most attention should be given to the following considerations, viz: Lapse of time since the field tests were conducted and the addition to certain pots of all ingredients which might prove beneficial other than the one supposed to be deficient. The field test upon the Wapping soil was conducted only one year and as long ago as 1891 and the last year of the field experiment upon the Limerock soil was 1893. The chief deficiency formerly noticeable upon these soils was potash, but nitrogen was needed to some extent. It is reasonable to suppose that recent cropping and management may have changed the conditions to such an extent that readily assimilable nitrogen, as was indicated by the pot experiment, is at present more deficient than potash.

The preliminary tests to ascertain the need of alkaline treatment led to the addition of calcium carbonate to the pots from which conclusions were to be drawn as to whether the supposed deficiencies existed. Other ingredients sometimes omitted in field experiments were also supplied. These additions were no doubt somewhat responsible for the exceptional results obtained in the pot experiment. Before experiments are conducted to ascertain food requirements of soils all other conditions should be made as favorable as economy will permit. It is obviously irrational to purchase plant food which may prove unnecessary after mechanical conditions, drainage, and the reaction of the soil are made favorable.

The referee on soil analysis has usually been expected to place samples of soil in the hands of the cooperating chemist a few months after receiving his appointment. It is obviously impossible under such circumstances to do more than canvass for soils upon which field experiments have been conducted, and which are thought, from the records at hand, to show a deficiency of some particular ingredient. Time scarcely remains for the referee to make a complete study of the detached field records, to say nothing of conducting experiments under his own supervision, which shall be especially designed to answer the particular question to be decided.

It seems probable, in view of the data presented in this paper, that much of the chemical work of this association has been done upon samples of soils which did not possess the characteristics attributed to them, and that hasty conclusions have therefore been drawn regarding the efficiency of certain methods. It seems too much to expect a method to answer questions regarding the assimilable nitrogen, phosphoric acid, or potash in a soil which needs draining, liming, or the addition of other ingredients aside from the so-called essential elements to put it in the best condition for the growth of most agricultural plants. Every crop the quantity of which is to be taken as a standard in judging the value of a general method for determining the particular requirements of a soil for assimilable nitrogen, phosphoric acid, or potash should have been grown after all general conditions of the soil had been made favorable for the full development of the plants.

SUMMARY.

The analytical results secured by this association have frequently failed to account satisfactorily in the case of given soils for differences caused by known fertilization and cropping.

This failure was probably due in many cases to the fact that the small amount of soil analyzed did not properly represent the large area from which it was taken.

Land upon which field experiments are to be conducted for showing its need of plant food should first receive such treatment, other than the application of the particular ingredient to be studied, as is economical and necessary for the proper growth of most agricultural plants.

Soils designed for use in testing methods for determining assimilable plant food should in many cases be subjected to pot experimentation before they are distributed by the referee.

Mr. Patrick. In view of the fact, as brought out in the paper just read, that a number of years elapsed between the liming of the fields and the taking of the samples, cropping had occurred right along, and perhaps some barnyard manure applied, it does not seem to me at all strange that the pot results did not agree with the field results. This paper enforces very strongly the teaching that the soils for experimentation should be taken from the field immediately after the crops are taken and the crop report is made from the field.

Mr. WHEELER. I think attention ought to be called to the fact that in the Limerock soil, which is supposed to be deficient in potash, no potash was subsequently applied and crops were grown continuously which would take out more potash. Notwithstanding these facts, we find that additional potash is not needed. It seems to me that the results point out the fact that we must be very careful about conducting our soil tests and know absolutely the deficiencies of the soil before we use it as a basis for comparing the merits of our analytical methods.

Mr. PATRICK. What the last speaker has said about the potash may be true; nevertheless, we observe in the first two soils that nitrogen was the thing that was needed. It was the presence of ammonium nitrate that brought out the fertility of that soil and not the addition of potash. Furthermore, we have always been taught to believe that soils develop available plant food in the course of years, especially when crops are grown upon them and the roots permeate the soil to a considerable extent, the roots not being easily removed with the crop.

Mr. HARTWELL. In order to bring the point once more before the association, let me say there was no attempt made to create a conflict between the pot and field experiments. We simply stated what the field experiments indicated at such a time and under certain conditions. The same crop was not tried in the pot experiments as was tried in the field experiments. The principal point is that we can not accept field results which were conducted some years previous as an indication of a lack of a particular ingredient in the soil at the time the sample is taken and distributed. Of course, it is well understood that one crop may cause a gradual deterioration in one ingredient of the soil and another may exhaust a different constituent.

Mr. SHUTT. I think one of the most important features of the referee's report is a certain suggestion which he made with regard to the statement of results of available plant food. He favors the idea of having them stated in pounds per acre. I think it a very wise suggestion to give the depth of the surface soil and also the weight of a cubic foot of the soil in stating these results. It would be unwise to adopt the method of stating the results as pounds per acre and reject that of the percentage of water-free soil, because the water content of a soil varies from day to day; it may be from 20 to 40 per cent of water would have to be taken into consideration when the weight of a cubic foot of soil was taken and the percentage of moisture would have to be calculated. The same is true with regard to the depth of the soil. We should have to reduce them all to the same depth as well as weight.

It seems wise to retain the plan we have now for stating the results on the water-free soil, and at the same time to accept the suggestion of making a statement in regard to the depth of the surface soil under consideration and its weight.

There is another question. The root systems of different plants have different depths, and consequently plant food available to one crop would be out of reach of another crop. It does not hold good in every case that we should state the amount of plant food available per acre. It seems to me that none of the results, as stated by the referee in this table, would justify us in excluding the citric acid method from consideration. Unfortunately, very few of the results have been carried through by the same analyst. There is really nothing which would prevent us from accepting the suggestion to state availability of plant food in the soil; nothing to prevent our accepting the citric acid method as being as accurate as the nitric acid, or fifth normal hydrochloric acid method. I certainly think that we have in a measure lost sight of what we started with in the estimation of available plant food. We have considered the soil instead of the plant. You will remember this whole affair started with the determination of the acidity of the plant-root sap. We supposed the plants received their nourishment from the soil through osmosis, consequently it seems to me the legitimate way would be to ascertain the foraging power of a plant in that connection. It is a matter of regret to me that the results published by Dr. Dyer in 1894, and which gave us the incentive, have not been corroborated or confirmed or duplicated on this side of the water. I have been using the citric acid method because there is really a scientific basis for it. I have used it continuously, and have never seen any scientific basis for the adoption of the ammonium chlorid, calcium chlorid, and other methods which have been introduced in the past few years. I should be sorry to see the citric acid method dropped. There is nothing to warrant such drastic measures.

Mr. Hartwell. I think the last speaker has failed to understand my position in regard to the question. I simply stated that the abandonment of the citric acid method had been recommended by former referees, and I also said that the results at present were not conclusive. I, for one, do not believe in dropping any of our methods which do not present any analytical difficulties, until we are very sure about the nature of our soil.

Mr. Dyer. I would like to claim your indulgence to say a few words on this subject; one of the greatest interest to me. I would like first to point out that in this series of analyses the 2 per cent citric acid solution has been used. That is not at all the same as the original 1 per cent citric acid method as proposed by myself. I should like to take this opportunity to make an explanation about that original process, and in regard to the way in which it has been applied by your association in some of the cooperative trials that have been made. My process was originally, and still is, the use of a 1 per cent citric acid solution, a definite volume being used for a definite quantity of soil, without relation to basicity of the soil in the matter of carbonate of lime. When I read my paper at the chemical society what seemed to me a very pertinent suggestion was made. It was that, as carbonate of lime neutralized citric acid, a sufficient quantity of acid should be used to neutralize the carbonate of lime in the soil and then enough citric acid should be present to leave the solution acid to the extent of 1 per cent. A great deal of work has been done since then by Dr. Woods, of Cambridge, on various soils in India using this process, and there were certain soils remarkably poor in phosphoric acid and some were undoubtedly poor in potash, as tested by field results, and in comparing these soils with other soils by the citric process these differences were not brought out as they should have been. He had used the modification which allowed for neutralization of the carbonate of lime. Those soils happened to be soils containing a good deal of chalk. It is the process as originally suggested in my paper, without allowing anything for carbonate of lime, which gave results according so beautifully with the field results that we might infer one from the other. He made what seemed a very pertinent suggestion. It was that solvents being the result of an attempt to do something that accorded with the sap acidity, it was right surely that the carbonate of lime should not be corrected. What I mean is this: If the plant gets its mineral food by means of the acidity of its sap, then an excessive quantity of carbonate of lime in the soil is a natural difficulty with which the plant has to cope. If we wipe out this difficulty by neutralizing the carbonate of lime, we are departing from the original principles of the process. Again, if we allow for the carbonate of lime we get a much larger quantity of citrate in the solution. Of course, whatever process is used must be an arbitrary one. The love of offspring is at once a virtue and an infirmity in that it is so strongly planted in the human breast. We never like to think that other peoples' children are better than our own until we have overwhelming evidence, and then we are obliged to give in.

I have a message to deliver to you, and a suggestion to make. Very shortly before his death Sir John Lawes, and also Sir Henry Gilbert, urged upon me strongly that if good fortune should bring me into contact with you in your annual convention I should make a strong recommendation on the subject of soil sampling. That is, as you have done so much work in the direction of bringing about uniformity of analyses, to see whether it is not possible for you to do something to bring about uniformity of soil sampling. In connection with some of the questions that have cropped up to-day my message comes in pertinently. I am afraid to say how many thousands of analyses we have made of certain soils. Almost every difficulty connected with soil sampling has necessarily been considered. In the earlier years of sampling at Rothamsted samples were taken of the surface soils without reference to the fact that the surface soil differs considerably in composition with each inch of depth. It is for this reason necessary that the sample should be taken in the shape of a cube or cylinder, all the cross sections of which shall have an equal area. Only when the sample is of this shape will the successive layers of soil be taken in their due proportion. Although attention has been very frequently directed to this matter, there is too much reason to suppose that not a little otherwise good soil work has been vitiated by reason of insufficient attention to the drawing of representative samples.

In 1856 the method of collection then and subsequently adopted was devised, and I, perhaps, can not do better than quote directly from Professor Warington's lecture (U. S. Department of Agriculture, Experiment Station Bulletin No. 8, 1892, p. 39). Professor Warington says:

> A frame made of stout sheet iron, in shape a rectangular prism, open at top and bottom, is driven into the soil by repeated blows of a wooden rammer till the soil has the same level inside and outside the frame. The soil inside the frame is then cut out and constitutes the sample of the first depth, or surface soil. That the frame is accurately emptied is ascertained by trials with a wooden gauge of the same depth as the iron frame. If a sample of the next depth is to be taken the soil is cleared away around the outside of the frame till the level is reduced to that of the bottom of the frame; the frame is then driven down again and the former operations are repeated.
>
> Soil sampling at Rothamsted is usually carried down to three depths, but in a good many cases it has been carried down to twelve depths. The area of the sampling frame used for the first depth is usually 144 square inches (12 by 12 inches), a smaller frame (6 by 6 inches) being used for the succeeding depths. * * *
>
> The iron frame has a stout rim along its upper edge to increase its strength. The best sampling frame is made of cast steel; this form of frame needs no rim. (Models of both the larger and smaller steel frames used at Rothamsted were on this occasion presented to the U. S. Department of Agriculture.)
>
> When the soil sampling is carried below the first depth care must be taken when digging around the frame that each depth of soil removed is placed by itself, so that when the pit is filled in the soil may be returned to its proper position. A record must be kept of the place where the sampling was conducted, as a soil can not be accurately sampled twice in the same place.
>
> Each sample of soil is weighed as soon as it is removed from the frame, and is put into a bag by itself. When the soil reaches the laboratory it is at once broken up by hand into small pieces, and laid on paper trays, which are placed on the shelves of a storeroom kept at a temperature of about 55° C. till thoroughly dry; each sample is then returned to its bag. This immediate drying of the soil at a low temperature is essential if changes in the organic matter, and especially nitrification, are to be stopped. This practice dates at Rothamsted from 1877. After drying the soil it may be stored till leisure is found for further work. Each bag is then weighed. The soil is crushed and passed through a $\frac{1}{4}$-inch sieve; the stones that do not pass through this sieve are weighed (and subsequently described) as stones. All that passes through the sieve is thoroughly mixed and a sufficient quantity is finely powdered for analysis. Mixed samples are prepared after the soil has passed through the $\frac{1}{4}$-inch sieve or after it has reached the stage of fine powder.

This method of taking soil samples, with the exception of the adoption of the definite temperature above mentioned for drying, has been, as has already been said, in use at Rothamsted since 1856, and it is interesting here to record that between 4,000 and 5,000 individual samples of the Rothamsted soils have been collected in the fashion above described.

The depth of 9 inches fixed for sampling the surface soil is somewhat greater than would now be adopted in the light of the experience that has been gained, although it would now be obviously inexpedient to make any change at Rothamsted, since in that case future samples would not be comparable with earlier ones. But in new soil work begun elsewhere both Sir John Lawes and Sir Henry Gilbert came to the conclusion that about 8 inches would be a fairer depth at which to mark off the surface soil from the subsoil. And since the metric system is so widely used in scientific work, the recommendation made by them to those initiating experimental soil work is to take the nearest round-number metric approximation and adopt a cube with the linear dimension of 0.2 meter as the unit for soil sampling. This would give to each layer of soil a depth of 7.874 inches, or practically 8 inches.

Of course, I can not make a recommendation, but I can ask you, if you think favorably of what I have said, to request some one to make a motion referring this question of uniformity of soil sampling, as well as methods of analysis, to a committee who should be empowered to thoroughly go into the question.

Mr. Macfarlane. I would like to express my thanks to Mr. Hartwell for having brought forward such a vast amount of information for us to digest. Generally speaking, his results seem to confirm a great deal of Wagner's teaching. We all know that Wagner has given us much information in regard to the application of fertilizers to soils, and has especially laid great weight upon the necessity for first attending to the physical and other conditions of the soil before we can expect that the consideration of its chemical constituents and the application of fertilizers will produce any great results. This point has been brought out by Mr. Hartwell in the pot experiments. It seems to me they settle in a great degree the question of plot versus pot in favor of the pot experiments. This belief was strengthened by my visit to Darmstadt during the past summer, where I saw Professor Wagner's pot experiments. I was very much impressed by these experiments, and the influence which a very slight addition of a fertilizing constituent has on the growing plant. There was one particular instance of the addition of a small amount of phosphoric acid to a certain pot which produced a very appreciable difference in the condition of the plant, and yet it would have been altogether impossible to have detected by analysis any additional phosphoric acid in the soil which was operated upon. That ought to be considered in studying the facts presented by Mr. Hartwell. I can only say that I have profited very much by the facts and figures which Mr. Hartwell has brought forward.

Mr. Ewell. I move that the referee on soils for next year be instructed to consider the adoption of methods for the mechanical analysis of soils and for the statement of the results thereof. I should like to enlarge that motion to include the method of sampling soil as suggested by Mr. Dyer.

The motion was referred to the committee on recommendations.

The report on liquor and food adulteration was then called for, but no report was presented, the referee not being present.

Mr. Winton. Before we leave this subject, which has been so thoroughly treated, I would like to say a word with reference to a division of the work on food adulteration. Anyone who has done work in this line appreciates the vast number of methods that must be employed. I have had some experience in the analysis of fertilizers and food products, and I think I can say, without exaggeration, that in the examination of food products with reference to adulteration the analyst is called upon to employ ten times as many analytical processes as in the examination of fertilizers. In addition to that, many of the methods which he uses are very little understood. There is a vast amount of work before us if we are to get methods in shape for the examination of food products. It is unfortunate that there is no referee at this convention on this important subject. The amount of work to be done is so large that it is a great pity we must lose the work of this entire year. To return to the suggestion that the work be divided—we might make a division of this work and the president be empowered to appoint a number of referees. That would accomplish the result, perhaps, but I would suggest that a better plan seems to be that we have but one referee, but that that referee be instructed to divide the work and to allot different sections to different men. By this means the whole field could be covered, and yet in a uniform way. These different subreferees could meet together under the general guidance of the referee and accomplish in a year a large amount of work. I will therefore introduce this resolution:

Resolved, That the subject of liquor and food adulteration be subdivided as follows: (1) meat and fish; (2) fats and oils; (3) cereal products; (4) infants' and invalids' foods; (5) saccharine products, including confectionery; (6) canned vegetables; (7) tea, coffee, cocoa, and the like; (8) spices and condiments; (9) vinegar; (10) flavoring extracts; (11) fruit products; (12) fermented and distilled liquors; (13) baking powder and baking chemicals; (14) preservatives; (15) dyes. That it be deemed the duty of the referee on liquor and food adulteration to associate with him such members of the association as he may see fit, each of whom shall be instructed to investigate a certain subject or subjects and prepare a report.

You will notice that the subject is divided into 15 sections, and perhaps you are all aware that there are only four or five food analysts in the association at the present time. From this it is evident that each one of these food analysts may get an appointment. The idea is that, although the number of workers is limited, the work can be satisfactorily carried on by giving each one a number of subjects; or, if a subject does not require especial investigation during a particular year, it can be kept out of sight entirely. I therefore submit this resolution.

Resolution seconded.

Mr. PATRICK. I wish to ask in what section, in that list of fourteen or fifteen subjects, do you place such articles as butter and cheese?

The PRESIDENT. May I suggest that the whole question be referred to a special committee to be reported upon at the afternoon session?

Mr. WILEY. I would like to suggest the advisability of including in that committee the referee for dairy products, so as to bring the whole matter under one head.

Mr. LEECH. That work is so extensive in itself it would seem worthy of having a separate referee. I agree with Mr. Winton that the entire food work is so broad that it should be subdivided. It is impossible for one person to cover the ground.

Mr. PATRICK. If I heard correctly, distilled liquors, wines, etc., were included. That is a rather large subject and one on which we already have a referee.

Mr. WINTON. I would state the reason for not including dairy products under this head. We examine dairy products with two objects in view: First, the detection of adulteration, and second, the determination of the nutrition contained in the milk. That is, it is not a question of adulteration alone, and for that reason only a part of the work would come under this head, and this was thought of in making the classification.

The resolution was referred to a special committee.

The PRESIDENT. We have already had the pleasure of a visit from the president of the American Chemical Society, and I see before me this morning the past president of the Association of American Agricultural Colleges and Experiment Stations. I am sure the association will be pleased to hear from Mr. Stubbs.

Mr. STUBBS. Mr. Chairman and gentlemen of this association: I am in mind of the time when I began to learn to swim. I had a fine teacher, consequently one without any heart, and when he threw me into the water he said: "There, swim!" Now, in the presence of this body of scientific men, whose work I appreciate as never before, whose influence upon the common welfare of society is already so great and yet destined to be so much greater, I feel that the platform is no place for me, but that I should be in the body of the audience listening. I am only sorry that duties pertaining to the work at New Haven made it impossible for me to get here in time for the work of this association beginning yesterday. I made all possible haste to get here in order that the Agricultural Experiment Station of Nevada might be represented, not worthily, but still represented.

I am very glad indeed that I belong to the ex's. I am reminded of a little story that is told of Senator Hoar. One of the ex-Senators called upon him at the Capitol, and having, of course, the privileges of the floor, went into the Senate Chamber and visited with him. As they were about to retire, the ex-Senator politely stepped back, saying: "After you, wise Senator." The grand old man from Massachusetts straightened up and replied: "Oh, no! the x's always go before the y's." Therefore, I am glad to meet you.

The PRESIDENT. We still have a few minutes before adjournment, and I will call for the report of the committee on recommendations.

Mr. WILEY. After we adjourned last evening the board of managers of the Cosmos Club extended the courtesies of the club to the members of this association during their stay in the city. We shall be glad to have you visit the club, where you will find papers, magazines, writing paper, etc., and something to eat and drink also, according to your taste, and where I hope you will be comfortable and where I know you will be welcome.

The report of the committee on recommendations was then read by the chairman, Mr. Huston.

REPORT OF THE COMMITTEE ON RECOMMENDATIONS.

NITROGEN.

(1) It is recommended that the neutral permanganate method (Street) be referred to the referee for 1901.

Adopted.

(2) It is recommended that the alkaline permanganate method (Jones) be referred to the referee for 1901.

Adopted.

PHOSPHORIC ACID.

1. On page 13, Bulletin No. 46, Division of Chemistry, U. S. Department of Agriculture, under the heading (b) Optional Volumetric Method. (1) Preparation of reagents, omit the following:

(*b*) Potassium nitrate or ammonium nitrate solution for washing. Dissolve 3 grams of the salt in 100 cc of water.

(*c*) Nitric acid solution for washing. Dilute 100 cc of nitric acid (specific gravity, 1.42) to 1 liter with water.

Adopted.

2. On page 14, Bulletin 46, under the directions for the determination of phosphoric acid (P_2O_5) by the optional volumetric method, after paragraph b, add: "b_2. Proceed as directed in b_1 with this exception, heat in water bath at 45° to 50°, add the molybdic solution, and allow to remain in the bath, with occasional stirring, for thirty minutes."

Adopted.

3. Following paragraph b_2 add: "b_3. Proceed as in b_1 to the point where the solution is ready to be placed in the water bath. Then cool solution to room temperature, add molybdate solution at the rate of 75 cc for each decigram of phosphoric acid present, place flask containing solution in shaking apparatus and shake for thirty minutes at room temperature, filter at once, wash and titrate as in preceding method."

Adopted.

Mr. Huston. The recommendation of Mr. Macfarlane would entirely change our methods for the determination of phosphoric acid, and that, of course, could not be done under the constitution without at least one year elapsing.

Mr. Wheeler's resolution is that we introduce a method for the examination of basic slag and make it a provisional method. The committee wishes to report that it recommends that the association instruct the referee to investigate the methods for the determination of availability in basic slag. It does not think it advisable at this time to adopt a provisional method.

Mr. Bartlett. We shall probably have some goods of that nature to analyze, and would it not be well to authorize us to use some method, perhaps that of simply determining the total phosphoric acid, as we do with bone meals?

Mr. Wheeler. I will state my reason for introducing that motion. Basic slag is now being shipped into California. It is also being shipped into the Southern States and is likely to be shipped into other States. It seems that in the interim which will elapse before we can investigate and adopt some method as official we might adopt the same method which prevails in Germany, so we can examine these goods by the same method they will be tested by before shipment here. In view of the fact that the 2 per cent citric-acid solution gives comparative results, I think we could safely adopt it if we so desire, not as an official method, but simply as provisional, until we can adopt something to which we can give our approval.

Mr. Macfarlane. I would like to say that I consider the recommendation of the committee eminently wise, and I hope Mr. Wheeler will not insist upon the adoption of a provisional method. The analyst should rest content with determining the total phosphoric acid, and let basic slag stand on its own merits.

Mr. Huston. I will say that we spent quite a long time considering the matter, and had many reasons for making this recommendation. In the first place, if you are to attach the same commercial value to available phosphoric acid in basic slags that you do to available phosphoric acid in other goods, the 2 per cent citric-acid solution gives a value to basic slag out of proportion either to the cost of production or its value in the field. I believe if you will thoroughly leach out basic slag with water you can remove a large portion of the free lime. With a more careful treatment with water you will reach a result on citrate soluble phosphoric acid nearer the commercial value and field value than by treating with a 2 per cent citric-acid solution. I believe it dangerous to adopt it as a provisional method at present. Pressure will be brought to bear to retain it as official. We ought to give the matter immediate and serious attention.

I think we would probably have to use more water and grind the material with the water, as in the method with acid phosphate. I believe a little manipulation of that sort will remove more of the free lime. Work in our laboratory shows that it takes out a large quantity.

There is another point in connection with availability, and that is the product of the reaction of neutral ammonium citrate on basic slag is not very soluble in the fluid itself, and where you take 100 cc of our 0.109 solution and treat 2 grams of slag with it you get a certain percentage soluble in that reagent. If you will add 400 cc of water, you will get a very much larger solubility, which convinces me that the product of the reaction is not soluble in the more concentrated fluid.

Mr. MACFARLANE. A word of caution in regard to the use of water in determining the available phosphoric acid. We have tried it and find it to be impracticable. You might, by moderate washing, say of several hours, increase the available phosphoric acid 1 or 2 per cent, but you can not by that means remove the free lime to the necessary extent. I could not recommend the chemists present to waste their time trying to remove all the free lime.

Mr. SHUTT. In this matter the agricultural chemist stands between the consumer and the vender. This basic slag is an imported article here, as well as with us. The universal method of analysis in Europe is the citric-acid method. When the goods come to be tested on this side we shall have to explain why our results do not coincide with those obtained by European chemists. The method seems to have some things in its favor.

Mr. HUSTON. If these goods come to the State of New Jersey, they will have to be treated at 40°. Our American manufacturers know what to expect when they ship goods to New Jersey. The foreign manufacturers will speedily become acquainted with the methods to be used on the basic slag, and will conform to our methods, or, at least, will know what to expect when the materials get here. I believe the citric-acid method gives results too high.

Mr. DYER. Are we not sometimes in danger of confusing two things, viz, availability and value? Phosphoric acid in two forms might be equally available ultimately, but the value might be different. It is well recognized, from the experiments of Wagner as well as other experimenters, that what we call the available phosphoric acid in slag has not the value to the farmer of a like quantity of phosphoric acid in the form of superphosphate. It might be equally available when the plant can get it. Surely the question of the value of a unit is a different question from availability.

Mr. Myers. It seems to me that this difficulty can be overcome by the State chemists, until they are ready to adopt some fixed policy, by treating basic slag about as they would floats or finely ground bone meal. It is a special product and has to be treated by a special method, and as a basis of valuation you can take the total phosphoric acid, and if the manufacturers want to claim more, then that is their misfortune or their good luck, as the case may be. At the same time the farmer knows that he is receiving a total quantity, which upon certain classes of land becomes rapidly available. There is no fraud practiced on anybody, and by changing the scale of valuation, for a valuation has to be fixed, as suggested by Mr. Dyer, the matter can be bridged over until such time as the association can establish a satisfactory method of analysis. I agree heartily with Mr. Huston that the method of analysis as adopted in Europe is not a satisfactory method. I think that the acid solution shows too much phosphoric acid. I do not think that is the method which will finally be adopted by this association, but I know of no method that can be offered now except, as Mr. Wheeler says, provisionally, and it is a question whether a provisional method, that may cause trouble later, ought to be put on the books. Anybody can determine the total phosphoric acid and report on that just as we do on total phosphoric acid in floats and in fine bone meal. It would be well also to note in the analysis the proportion of finely ground meal in the Thomas phosphate, because the availability of Thomas phosphate is undoubtedly affected very largely by the fineness of the powder in which it is presented to the farmer.

Mr. Wheeler. I think the members of the association are aware that there is a great difference in the assimilability of the phosphoric acid in basic slag. The slags, which contain a considerable amount of silica, are much more assimilable than those where a less quantity of silica is present. I would like to ask the gentlemen of the committee whether they can tell us positively that with two classes of slag the one containing less silica may be finer than the other and still be less assimilable? It seems to me the answer to that question should be a positive one.

Mr. Huston. We have no American data on the subject.

At this point the meeting was adjourned to meet at 2 o'clock.

SATURDAY—AFTERNOON SESSION.

The meeting was called to order by the president at 2 o'clock.

The PRESIDENT. We will now have the report on ash.

REPORT ON ASH.

By A. E. SHUTTLEWORTH, *Referee.*

The following circular letter and outline of the work proposed were sent to a number of American chemists most likely to cooperate with us:

MY DEAR SIR: At its recent meeting in San Francisco, the Association of Official Agricultural Chemists, considering the subjects of soil and ash analysis of sufficient importance, provided for the appointment of separate referees.

Dr. G. S. Fraps, of the North Carolina College of Agriculture, appointed associate referee of ash analysis, and myself, appointed referee, have pleasure in inviting you to assist us in this important work by conducting one or more of the investigations as outlined on inclosed sheet.

Upon the receipt of your reply informing me that you will join in this work, and indicating the investigations that you have chosen, I will immediately express to your address the necessary samples and forms.

Yours, very truly, A. E. SHUTTLEWORTH.

PROPOSED RESEARCH WORK.

1. (*a*) Make by the official method (see "Methods of Analysis," 1899, p. 77) at least three crude ash determinations in sample No. 1, using for each 12, or approximately 12, grams of the material.
   (*b*) Determine the carbon and the carbonic acid in this crude ash, reporting on Form I, per cent of crude ash, per cent of carbon and carbonic acid in crude ash, and per cent of carbon-free ash, which is the crude ash less the carbon and carbonic acid.
2. (*a*) In each separate ash prepared under 1, determine the total silica (i. e., make no separation of crude silica into soluble and insoluble), reporting on Form II crude silica in carbon-free ash.
   (*b*) In sample marked No. 2 make three total silica determinations similar to (*a*) under 2, reporting results on Form II.
3. (*a*) Determine by the official method the per cent of K_2O in each sample of ash prepared under 1, reporting on Form III per cent of K_2O in carbon-free ash.
   (*b*) In sample 2 make three K_2O determinations similar to (*a*) under 3, reporting results on Form III.

Notes.—1. In each determination, (*a*) and (*b*) of 2, not less than 1 gram of the ash is to be used, and the filtrates from these may be employed respectively for (*a*) and (*b*) under 3.

2. To determine carbonic acid see 6 on p. 79 of "Methods of Analysis," 1898.
3. In (*a*) and (*b*) of 2 collect in a tared Gooch crucible and dry to constant weight at 110° C. After incineration the loss in weight gives the carbon.
4. Results are requested on or before July 1.

REMARKS.

Eight chemists replied that they would undertake the work, and asked that samples be forwarded to them. The samples were forwarded on March 30, together with the following letter of explanation and blank forms for reporting results:

MARCH 30, 1900.

DEAR SIR: In response to your letter of recent date, I am sending to you by to-day's express samples marked No. 1 and No. 2 for A. O. A. C. work, as outlined in my circular letter already received by you.

In calculating your results, please observe that sample No. 1, which is prepared from oat straw, contains of water-free—that is, of dry substance—95.15 per cent.

Sample No. 2, which is ash prepared from the same oat straw by the use of calcium acetate solution in my platinum ash apparatus (see pp. 304 and 305 Experiment Station Record, Vol. XI, No. 4), contains 86.61 per cent carbon-free ash. Please calculate results on the basis of 86.61 per cent carbon-free ash. The remaining 13.39 per cent are composed of the following:

	Per cent.
CaO from added calcium acetate solution	2.50
CO_2 from added calcium acetate solution	10.15
Carbon from added calcium acetate solution	.74

We hope to ascertain by these tests whether the ash which you prepare from sample No. 1 by the official method contains the same percentage of SiO_2 in the carbon-free ash as No. 2. These tests are to see whether ash prepared by the official method is quite decomposed by hydrochloric acid or not. If it is not, then SiO_2 determinations are too high, owing probably to chemical combination of SiO_2 and bases during incineration.

We hope further to ascertain whether part of the potash is either volatilized or united chemically with SiO_2 by incinerating to an ash.

Inclosed please find forms upon which please report your result.

I am, very respectfully yours,

A. E. Shuttleworth.

RESULTS REPORTED.

Owing chiefly, I suppose, to pressure of work, only three of those to whom samples were sent reported results. These are Dr. G. S. Fraps, assistant chemist North Carolina College of Agriculture and Mechanic Arts, Raleigh, N. C.; Mr. C. C. Moore, of the U. S. Department of Agriculture, Division of Chemistry, Washington, and William P. Gamble, B. S. A., special assistant in the department of chemistry, Ontario Agricultural College, Guelph, Canada. To these gentlemen we are indebted for the valuable results contained in the following tables:

Table I.—*Carbon-free ash in sample No. 1.*

Analyst.	Determinations.	Percentage in water-free substance.			
		Official method.		New method.	
		Single determination.	Average.	Single determination.	Average.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Moore, C. C.	1	6.25	6.25	....	....
	2	6.25			
Fraps, G. S.	1	5.56	5.55	....	....
	2	5.59			
	3	5.49			
Gamble, Wm. P.	1	6.40	5.87	....	....
	2	5.01			
	3	6.20			
	4	5.66	5.655	....	....
	5	5.65			
	6	....	....	6.38	[1]6.38
	7	....	....	6.39	
	8	....	....	6.36	

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[1] Prepared from sample No. 1 by the addition of calcium acetate in a closed platinum apparatus.

Crude ash of vegetable and animal substances, ever so carefully burned, contains varying quantities of carbonic acid, carbon, and foreign matters. Therefore percentages determined and stated as crude ash may vary as much as 0.2 to 2 per cent in the same substance. Because of this I think chemists should at least make carbonic acid and carbon determinations in the crude ash, and state percentages as carbon-free ash. Even with the greatest care, employing the official method for the preparation of the ash, a lower percentage of carbon-free ash will be obtained than that actually contained in the substance, owing to losses of chlorids and probably more or less phosphorus and sulphur by volatilization. Of the correctness of this fact I am convinced by investigations which have been made by me and published in my inaugural dissertation under the title, "Eine neue Methode der Aschenbestimmung." This fact is also confirmed, as shown in Table I, by the work of Messrs. Moore, Fraps, and Gamble. The average percentage of carbon-free ash, determined in sample No. 1 by what I have called a new method, is from 0.13 to 0.83 per cent higher than the averages determined in the same sample by the official method. Why should the individual percentages of carbon-free ash in the same substance and determined by the same chemist with undoubted care vary all the way from 5.01 to 6.4 per cent? Again, why should the averages of carbon-free ash determined in the one substance by different chemists working equally carefully vary from 5.55 to 6.25 per cent? These variations, I believe, are due to volatilization of varying quantities of certain ash constituents. By the new method, which employs a closed platinum apparatus and calcium acetate solution, individual determinations of percentages of carbon-free ash in the same substance are concordant to the first decimal, as shown by determinations 6, 7, and 8 in Table I.

TABLE II.—*Crude silica in carbon-free ash.*

Analyst.	Determinations.	Percentage in ash from sample No. 1.		Percentage in sample No. 2.	
		Single determination.	Average.	Single determination.	Average.
Moore, C. C	1	14.13	14.125	15.27	15.23
	2	14.12		15.19	
Fraps, G. S	1	15.81	15.84	15.44	15.56
	2	15.89		15.33	
	3	15.82		15.91	
Gamble, Wm. P	1	15.93	19.58	15.50	15.39
	2	26.72		15.34	
	3	16.08		15.35	
	4	15.35	15.45		
	5	15.54		....	....
	6	....	....	15.24	[1] 15.22
	7	....	....	15.23	
	8	....	....	15.19	

[1] These determinations were made in an ash prepared from sample No. 1, but the ash was prepared by Mr. Gamble exactly as in sample No. 2.

Heat employed in the reduction of substances to ash very readily causes more or less fusion, whereby the bases of the ash enter into chemical combination with the silica forming a product or products which resist the action of hydrochloric acid, and are therefore collected and weighed in ash analysis as silica. These circumstances may and do frequently result in very erroneous silica determinations Take, for example, Mr. Gamble's second determination, 26.72 per cent, Table II, as compared with the other determinations of crude silica. It is 10.64 per cent greater than the next highest. This difference, I am confident, is not due to errors in work, but to fusion with the formation of products not decomposable in hydrochloric acid. In preparing the ash (Tables I and II, Gamble) from which this silica was separated the heat rose a little above dull redness, which immediately caused distinct fusion in the ash.

Sample No. 2 is an ash prepared by the use of calcium acetate in my closed platinum apparatus. The use of calcium acetate solution in moistening the substance before reducing it to an ash appears to prevent the formation of any trace of indecomposable products in the ash. Consequently, it was expected that not only would the individual determinations of crude silica in sample No. 2 be more uniform, but that the average percentage would be lower than in the ash prepared from sample No. 1 by the official method. With the exception that one chemist found more silica in sample No. 2 than in the ash of sample No. 1, the figures of Table II justify the above expectations.

TABLE III.—*Potash* (K_2O) *in carbon-free ash.*

Analyst.	Determinations.	Percentage in ash from sample No. 1.		Percentage in sample No. 2.	
		Single determination.	Average.	Single determination.	Average.
Moore, C. C	1	28.27	28.19	30.87	30.96
	2	28.10		31.05	
Fraps, G. S	1	32.46	32.51	32.09	....
	2	32.85			
	3	32.22			
Gamble, Wm. P	1	31.59	29.57	32.19	32.25
	2	27.72		32.27	
	3	29.41		32.29	
	4	31.21	31.09		....
	5	30.96			
	6	....	....	32.75	32.86
	7	....	....	32.87	
	8	....	....	32.96	

If fusion in the preparation of ash forms indecomposable products, lower percentages of potash might be expected to accompany higher percentages of silica. That this is the case in a number of instances the figures in Tables II and III show. In addition to this tendency of fusion to lower the percentages of potash, volatilization of chlorids in the preparation of an ash acts in the same direction. These two circumstances account for the higher percentages of potash in sample No. 2 than in the ash of sample No. 1. The tendency of fusion and volatilization to lower the percentage of potash is made very evident by Gamble's determination (2). That a somewhat higher temperature than that of dull redness was allowed in preparing that ash, and that more or less fusion occurred in the ash, are facts known to me. Table I shows that Gamble's determination (2) is the lowest percentage of carbon-free ash; Table II shows that it is the highest percentage of crude silica, and Table III that it is the lowest percentage of potash.

All chemists will agree with me that the place to begin improving the method of ash analysis is in the preparation of the ash. I am of the opinion that volatilization and fusion in its preparation are two important sources of error; that the use of calcium acetate solution overcomes the difficulty of fusion, and that the use of a closed platinum apparatus prevents volatilization.[1]

In conclusion, allow me, on behalf of this association, to thank those chemists who have furnished me with analytic results, and also allow me to specially mention the heartiness of the cooperation of the associate referee, Dr. G. S. Fraps, of Raleigh, N. C.

The referee recommends the use of calcium acetate and burning ash in some way to prevent volatilization, which does occur, even with great care, by the official method.

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[1] See Twenty-fifth Annual Report of the Ontario Agricultural College and Experimental Farm, 1899, Toronto, Ontario, pp. 42–43.

The president called for any papers bearing on the subject of ash determinations, Mr. Fraps presented one, as follows:

REPORT ON LOSS OF SULPHUR IN PREPARING ASH OF PLANTS.

By G. S. Fraps.

It is becoming generally known that the sulphur contained in an ash does not necessarily represent the sulphur content of the plant. Berthelot[1] states that the determination of phosphorus and sulphur when the plant is burned to an ash is often incorrect, and discusses the conditions theoretically necessary that no loss take place. Dr. Wiley[2] says, "Unless special precautions be taken, however, a portion of the organic sulphur and phosphorus may escape as volatile compounds during the combustion," and refers to the method of Berthelot and André for determining total sulphur in soils, viz, oxidation in oxygen and passing the vapors over hot alkaline carbonates. S. Bogdonow[3] states that the estimation of the sulphur content of a plant by determining the sulphur in the ash is incorrect. He determines the sulphur in the plant preferably by the method of fusing it with caustic potash and potassium nitrate. Comparing his analyses of cereals made by this method with Wolf's tables of ash analyses, he concludes: (1) The sulphur in the ash does not give even an approximate idea as to the sulphur in the plant; (2) Plants contain considerably more sulphur than has been supposed; (3) The sulphuric acid of the soil is of practical importance. It may be added that he found fertilization with sulphates advantageous to certain Russian soils.

The following experiments were made to determine whether or not sulphur is lost during the incineration of vegetable substances. No case was found in which all the sulphur of the plant was contained in the ash. The determination of the sulphur was made by two methods: (1) Ten grams were burned to an ash at as low a temperature as possible, and the sulphur determined in it; (2) Ten grams of substance were burned with the addition of 20 cc of a solution of calcium acetate containing 29.2 grams per liter, as recommended by Mr. A. E. Shuttleworth. The results were as follows:

Sulphur found in plants.

Substance.	Burned with calcium acetate.	Alone.	Loss.
	Per cent.	*Per cent.*	*Per cent.*
Oat straw	0.158	0.151	4
Crimson-clover hay	.173	.137	28
Green rape	.503	.471	6
Wheat bran	.055	.000	100
Corn silage	.098	.082	16
Timothy hay	.085	.076	11
Cotton-seed meal	.222	.071	68
Soy beans	.161	.091	58
Linseed meal	.091	.038	58

The sulphur lost when the material was burned by itself is from 6 to 100 per cent more than when burned with calcium acetate, and it has not been proved that the calcium acetate retains all the sulphur.

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[1] C. r.: 128, 17.

[2] Principles and Practice of Agricultural Analysis, Vol. III, p. 37.

[3] Jour. russ. phy-chem. Ges.: 31, 471.

The differences in the above table may in some cases seem insignificant, but owing to the quantity of material employed it requires 0.0007 gram of barium sulphate to produce a difference of 0.001 per cent. It is plain that the determination of sulphur in an ash prepared in the usual way is far from giving any idea as to the sulphur in the plant. Any conclusions drawn from such analyses are liable to prove erroneous.

The president here called for the report of the committee on nominations, and the following names were presented by the chairman, Mr. Huston, as officers of the association for the ensuing year:

REPORT OF COMMITTEE ON NOMINATIONS.

For president, L. L. Van Slyke, Geneva, N. Y.

For vice-president, H. J. Wheeler, Kingston, R. I.

For secretary, H. W. Wiley, Washington, D. C.

For additional members of the executive committee, W. R. Perkins, of Agricultural College, Mississippi, and F. W. Traphagen, of Bozeman, Mont.

The secretary of the association was instructed to cast the ballot of the association for the several nominees.

The report of the committee on recommendations was then called for, and was presented, as follows:

REPORT OF COMMITTEE ON RECOMMENDATIONS.

SOIL.

(1) It is recommended that determinations of phosphoric acid be made by N/5 nitric acid as well as by N/5 hydrochloric acid.

Adopted.

(2) It is recommended that a 3-mm sieve be adopted where the soil is to be used for determinations requiring 100 grams or more.

Mr. Hartwell. I would like to say just a word about the reason for recommending a 3-mm sieve. I found that this size was commonly used in Germany, and it was stated to me that sometimes material would not pass a 2-mm sieve which should be subjected to the action of the solvent. Personally I am not particular whether a 2-mm sieve or a 3-mm sieve is used, but I think we should have a larger sieve for this work.

Recommendation adopted.

(3) The referee's report should contain such data as are necessary for converting the percentage of an ingredient into the number of pounds of the same in a given area of soil at the depth sampled.

Adopted.

The motion of Mr. Ewell, that the referee on soils for next year be instructed to consider the adoption of methods for the mechanical analysis of soils and for the statement of the results thereof, was adopted. The second part of the motion, viz, that the referee on soils for the next year be instructed to consider the adoption of a method for the sampling of soils in accordance with the suggestion of Mr. Dyer, was treated separately and was also adopted by vote of the association.

Mr. Huston. There was another matter referred to this committee, but for which we have no papers. It was the motion of Mr. Wheeler in regard to the Gooch-Austin method of determining phosphoric acid. The motion is that the method be referred to the referee on phosphoric acid for the ensuing year.

Adopted.

REPORT OF COMMITTEE ON SUBDIVISION OF FOOD AND LIQUOR ADULTERATION.

Resolved, That it be made the duty of the referee on liquor and food adulteration to associate with him such members of the association as he may see fit, each of whom shall be instructed to investigate and report on one or more of the following subjects:

(1) Meat and fish.
(2) Fats and oils.
(3) Cereal products.
(4) Infants' and invalids' foods.
(5) Saccharine products, including confectionery.
(6) Vegetables, canned, dried, or otherwise preserved.
(7) Tea, coffee, and cocoa.
(8) Spices and condiments.
(9) Vinegar.
(10) Flavoring extracts.
(11) Fruit products.
(12) Fermented and distilled liquors.
(13) Baking powder and baking chemicals.
(14) Preservatives.
(15) Dyes.

Mr. Patrick. I suggest that if the dairy products, butter and oleomargarine, are not to be included under the head of oils, it would be well to specify that. Otherwise there might be a misunderstanding. The referee, not being here, might understand that the oils would include the butter fats, oleomargarine, etc.

Mr. Winton. The report is certainly a little indefinite in that regard, but perhaps it is sometimes a virtue not to have things too exact. If the analyst were working on lard and olive oil his work would lead him to try the same method on oleomargarine and dairy products; it is very hard to draw a sharp line. I do not think there is any danger of our referees quarreling as to who shall have the privilege of doing the most. However, I appreciate the inconsistency noted and shall be perfectly agreeable to any amendment.

Mr. Patrick's amendment seconded.

Mr. Huston. It would be safer if you made the amendment positive instead of negative. Perhaps it would be better to define what the referees must do.

Mr. Winton. I think that point could be met by adding a word or two in connection with dairy products in another place. That is to say, the referee on dairy products may be instructed to consider the matter of adulteration in connection with his work.

Mr. Van Slyke. There are in the various States men who are charged with the enforcement of certain laws, and these gentlemen, as some of us have had occasion to know, are very much stirred up when the word oleomargarine or anything of that sort is mentioned in connection with dairy products, and I think we ought to be somewhat cautious about placing oleomargarine on our records as a dairy product. Between us it is all right, but we do not want to put ourselves on record and have these gentlemen point at us and say that the association classes oleomargarine and similar adulterants as dairy products.

The recommendation on subdivision of foods was referred back to the committee for exact statement.

The president then called for the report on foods and feeding stuffs, which was presented by Mr. Krug.

REPORT ON CATTLE FEEDS.

By William H. Krug, *Referee.*

The present report covers the work of two years, and it has been thought best by the referee to consolidate the results obtained on the samples sent out in 1899 and 1900, respectively, as the instructions with reference to the investigation of certain doubtful features in the analytical methods underwent no change. Early in 1899 Mr. Smith, at that time referee, sent out three samples, consisting of wheat, bran, and peas, respectively. Owing to the fact that the sample of peas was reported to contain very little galactan, it was thought advisable, when it was found that no report would be made at the San Francisco meeting, to send out a sample of clover seed and an additional sample of bran. In the tables which accompany this report the bran sent out in 1899 is marked No. I, and that sent out in 1900 No. II. The instructions which accompanied the samples followed the lines of investigation suggested by the referee in 1898. Subsequently both Mr. Smith, the referee, and Mr. C. A. Browne, jr., the associate referee, found that they would not be able to complete the work, and at the request of the president of the association I have undertaken the the compilation of the results. In answer to the circular letter sent out by Mr. Smith thirty chemists requested samples, of whom only eight have returned reports.

THE ESTIMATION OF MOISTURE.

It being evident that variations in the amount of moisture found in the samples would seriously affect the results when calculated to a dry basis, the referee for 1899 requested that this determination be made in all samples. The following results have been obtained:

Table I.—*Moisture determinations.*

Analyst.	Wheat.	Bran I.	Bran II.	Peas.	Clover seed.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Hatch Experiment Station	11.98	10.20	10.02	10.93	7.05
G. S. Fraps, North Carolina College of Agriculture	....	11.41	13.00	....	....
H. N. Grettenburg, Iowa Experiment Station	7.60	7.48	10.13	8.65	5.03
C. A. Browne, jr., Pennsylvania State College	11.37	9.75	11.52	....	7.16
C. P. Beistle, Pennsylvania State College	11.44	9.98	12.26	....	7.77
L. H. Van Wormer, Michigan Experiment Station	12.32	....	....	....	8.38
John Stewart, Utah Experiment Station	7.14	6.72	....	....	....
O. W. Knight, Maine Experiment Station	12.59	....	12.41	11.68	....
W. H. Krug, United States Department of Agriculture	12.68	11.47	11.85	....	10.37

COMMENTS.

G. S. Fraps.—Moisture was determined by the official method in a Caldwell water oven and drying tubes. The temperature in the oven was 98° C.

C. A. Browne, jr.—Determinations made at close of work, two weeks later than those made by Mr. Beistle. Bottles kept corked except when weighing samples for analysis. Samples dried in a bath at the temperature of boiling water.

C. P. Beistle.—Determinations made at beginning of work, immediately after opening bottles. Bran sample very moist.

(Note by referee: The remarks of Mr. Browne and Mr. Beistle apply to Bran No. II and the sample of clover seed.)

John Stewart.—Caldwell's hydrogen drying oven was used and duplicate determinations were made according to the official method.

DISCUSSION OF RESULTS.

Mr. Stewart also determined the moisture in the sample of wheat and in bran No. I by drying twelve hours at 98° C., which temperature is from 1.5° to 2° higher than the boiling point of water, at the station where he is located. He found that by this method the wheat contained 12.68 per cent and the bran No. I, 10.95 per cent of moisture.

The results stated by me were obtained by drying five hours in vacuo at 100° C.

The table shows that with a few exceptions there is absolutely no uniformity in the amount of moisture reported by the different chemists. I believe that this is chiefly due to the fact that the time specified by the method is not sufficient to remove all the moisture. It has been my experience repeatedly that when substances are dried in vacuo at 100° C. they continue to lose weight for about twelve hours, after which time either a slight increase occurs or the weight remains practically constant. I have found this to be true not only of such materials as fodders and cereals, but also of the comparatively thin films obtained in the analysis of tanning materials. It would seem rational to suppose that in the latter case, when a large surface is exposed, the moisture would be driven off very readily, but this does not appear to be so, a small fraction of the water, amounting usually to from 1 to 2 per cent, being retained very stubbornly and requiring from five to seven hours additional drying. It is evident, on the basis of the results shown in the table, that additional work on the determination of moisture is necessary, the purpose of such work being to fix the time limit and the exact temperature. The latter point would appear to be one of little importance, but it must be remembered that the boiling point of water is rather an uncertain quantity, and may vary from 1 to 2 degrees, depending upon the altitude and atmospheric conditions.

THE ESTIMATION OF STARCH.

Acting upon the suggestions made in 1898, the provisional diastase method and the influence which the solubility of the pentosans may have on the final result have been further investigated.

The following results have been obtained in the estimation of starch:

TABLE II.—*Results obtained with the diastase method.*

Analyst.	Wheat.		Bran I.		Bran II.	
	In original material.	Calculated to dry basis.	In original material.	Calculated to dry basis.	In original material.	Calculated to dry basis.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Hatch Experiment Station	52.36	59.49	24.61	27.41	28.60	31.78
H. N. Grettenburg, Iowa Experiment Station	[1]25.96	....	24.83	26.82	[1]20.11	....
C. A. Browne, jr., Pennsylvania State College	58.99	66.55	28.75	31.85	28.99	32.90
C. P. Beistle, Pennsylvania State College.	59.20	66.84	29.83	33.14	27.68	31.41
John Stewart, Utah Experiment Station.	60.50	65.15	....	....	....	....
O. W. Knight, Maine Experiment Station	56.86	65.04	....	....	30.04	34.31
W. H. Krug, U. S. Department of Agriculture	55.71	63.79	27.00	30.49	28.11	31.89
Averages	57.27	64.47	27.00	29.94	28.68	32.46

[1] Excluded from the averages.

COMMENTS.

E. B. Holland.—Wheat and bran No. I: Residues practically free from starch. Both solutions gave precipitate with phloroglucin after regular treatment. Malt solution prepared in the old way by allowing to stand overnight. Bran No. II: The copper oxid was collected in sugar tubes and reduced in a current of hydrogen. The residues, examined with iodine under a microscope, were free from starch. The malt solution was prepared as follows: Twenty-five grams of fresh-ground malt were treated overnight with 500 cc of water in a flat liquor flask, which exposed a large surface, filtered, and 40 cc added to the gelatinized starch. Blanks were made on 32 cc, which allowed a direct subtraction of the equivalent dextrose. One of the most prominent chemists in this section, using large quantities of malt, states explicitly that malt should be ground immediately before use, as it rapidly loses strength afterwards.

C. A. Browne, jr.—Bran No. II: Copper oxid filtered in a tube, oxidized in a current of oxygen and weighed as CuO. Directions prescribed in Bulletin 46 were followed except as regards neutralization. After inversion with hydrochloric acid, sodium hydroxid with a drop of phenolphthalein was used instead of sodium carbonate. The troublesome frothing incident to the use of sodium carbonate was thus avoided, and comparative determinations have shown no difference in the results.

C. P. Beistle.—Copper oxid filtered in tube, reduced in a current of hydrogen and weighed as copper.

John Stewart.—Duplicates on the sample of wheat differed by a little more than 1 per cent. The diastatic fermentation continued twenty-four hours, and even then the microscope revealed insignificant traces of starch in some of the residues. Owing to the absence of convenient means of electrolyzing the copper, I used the potassium cyanide method.

O. W. Knight.—The precipitate was dried at 100° and weighed as Cu_2O, the copper being calculated therefrom. Correction was made for the malt used. A portion treated as for the starch determination gave no pentose bodies either in the bran or wheat.

DISCUSSION OF RESULTS.

The results obtained are rather unsatisfactory with reference to the sample of wheat. The most concordant results were obtained on bran No. II. The fact that such satisfactory figures are reported on bran No. II renders it rather difficult to explain the fluctuation noticeable in the samples of wheat. To some extent this may be due to the different methods used for determining the copper, though this could hardly cause a difference of 8 per cent, such as exists between the lowest and highest results reported. I am rather inclined to believe that the trouble lies in the failure to follow the method strictly, especially since I find that in all cases where the analyst has reported duplicates these agree very well, showing that the analyses have been made in a uniform manner. Naturally slight differences in the manner of using this method would not be so apparent in a material containing comparatively little starch, such as a bran, but would be much more in evidence in the analysis of a cereal. Another cause of error is to be sought in the small amount of the original material which enters into the final calculation, this being only 0.12 gram. Thus a variation of 1 milligram in the amount of copper obtained causes a difference of about 0.4 per cent in the amount of starch found. This forcibly demonstrates that extreme accuracy in the application of the method is essential.

Mr. Grettenburg, of the Iowa Experiment Station, reported comparative results obtained by the diastase, Maercker, and salicylic acid methods, which are given in the following table:

Table III.—*Results obtained by the diastase, Maercker, and salicylic acid methods.*

Method.	Wheat.	Bran I.	Bran II.	Peas.	Clover seed.
	Starch.	Starch.	Starch.	Starch.	Starch.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Diastase method	25.96	24.83	20.11	58.56	22.37
Maercker method	32.93	19.98	28.05	50.90	21.49
Salicylic acid method	33.01	25.20	26.86	58.49	21.85

In consideration of the fact that both the Maercker and salycylic acid methods uniformly give higher results owing to the hydrolysis of the pentosans present, the data reported here are, on the whole, so conflicting as to permit of no explanation.

Mr. C. A. Browne, jr., investigated the applicability of taka-diastase as a substitute for the malt solution, and obtained the following results on the sample of wheat and on bran No. I.

Table IV.—*Results obtained with taka-diastase.*

Material.	In original material.	Calculated to dry basis.
	Starch.	Starch.
	Per cent.	*Per cent.*
Wheat	53.66	60.54
Bran No. I	25.27	28.00

The time of digestion in these analyses was twelve hours, and a blank showed no reducing substance. Mr. Browne concludes that taka-diastase is not a suitable substitute for the malt solution.

In 1898 it was suggested by me that the hydrolytic effect of the malt solution on the pentosans present be fully investigated, since at that time there was quite a difference of opinion on this point between chemists. It is unfortunate that this matter has been practically neglected so far as comparative results are concerned, since it can hardly be called a problem difficult of solution.

Mr. Holland reports that a pentosan determination carried out on 78 cc of the solution obtained with bran No. II yielded 0.0065 grams of phloroglucid, a weight too small to admit of the application of the usual formula of calculation.

Mr. Browne, jr., reports that the malt solution dissolved 2.63 per cent of the pentosans present in bran No. II.

The solubility of the pentosans has been thoroughly investigated by me this year. The first object in view was to determine the extent to which the pentosans would be hydrolyzed by the diastase method. For this purpose 3 grams of the sample of wheat, of bran No. I and of bran No. II were gelatinized, treated with 25 cc of malt solution until the residue was free from starch, cooled, made up to 250 cc, filtered, and 100 cc evaporated to a small volume, in which the pentosans were determined in the usual manner. The following results were obtained:

Table V.—*Solubility of the pentosans in the estimation of starch by the diastase method.*

Wheat.		Bran I.		Bran II.	
Wt. phloroglucid.	Pentosans dissolved.	Wt. phloroglucid.	Pentosans dissolved.	Wt. phloroglucid.	Pentosans dissolved.
Gram.	*Per cent.*	*Gram.*	*Per cent.*	*Gram.*	*Per cent.*
.0216		.0208		.0204	
.0212		.0215		.0218	
.0214					
.0212					
[1].02135	[1].208	[1].02115	[1].191	[1].0211	[1].186

[1] Average.

A blank determination with the malt solution showed that this contained no pentosans, and the results indicated that no appreciable amount of the pentosans present in the materials examined had been hydrolyzed. It was then thought advisable to investigate the effect of the time element on the solvent action of the malt solution, the data thereby obtained being, furthermore, confirmatory of those just given. Since starch and various sugars, when distilled with 12 per cent of hydrochloric acid, yield small quantities of volatile products precipitable by phloroglucol, it was thought best to work with a material both rich in pentosans and free from starch. For this purpose a considerable quantity of bran was very finely ground, boiled with water until all the starch was gelatinized, cooled to 55° C., and treated with malt solution until all the starch appeared to be converted. It was then washed thoroughly and again treated with the malt solution at 55° C. for three hours. The residue was again thoroughly washed, boiled with 1 per cent hydrochloric acid for about 15 minutes, neutralized with sodium carbonate, and washed with distilled water until the wash water showed no traces of chlorids. The dried residue was free from starch and was found to contain 33.61 per cent of pentosans. Three grams of this bran were treated exactly as previously described. The time of action varied from one-half to two hours.

Table VI.—*Effect of time on the solubility of the pentosans in the estimation of starch by the diastase method.*

One-half hour.		One hour.		One and one-half hours.		Two hours.	
Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.
Gram.	*Per cent.*	*Gram.*	*Per cent.*	*Gram.*	*Per cent.*	*Gram.*	*Per cent.*
.0140	....	.0176	....	.0117	....	.0177	....
.0132	....	.0156	....	.0105	....	.0186	....
.0144	....	.0143	....	.0110	....	.0203	....
.0140	....	.0151	....	....	....	.0193	....
[1].0139	....	[1].0157	....	[1].0111	....	[1].0189	....

[1] Average.

In every case the amount of phloroglucid was too small to permit the application of the usual formula for calculation to pentosans, and the results are sufficient evidence that during the estimation of starch by the diastase method the error caused by the hydrolysis of the pentosans may be safely disregarded. They also indicate that at least a portion of the volatile products precipitated by phloroglucol are directly derived from the conversion products of the starch itself.

Another question which now arose was as to whether the hydrolysis of this small quantity of pentosans is due to the direct action of the diastase or is caused by the water.

To determine this point 3 grams of the same bran, free from starch, were digested exactly as previously described, the addition of 25 cc of malt solution being omitted. The time of action varied from one-half to two hours. The results are given in the following table:

Table VII.—*Hydrolysis of the pentosans by the water in the estimation of starch by the diastase method.*

One-half hour.		One hour.		One and one-half hours.		Two hours.	
Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.
Gram.	*Per cent.*	*Gram.*	*Per cent.*	*Gram.*	*Per cent.*	*Gram.*	*Per cent.*
.0068	....	.0170	....	.0124	....	.0137	....
.0064	....	.0149	....	.0161	....	.0157	....
.0100	....	.0138	....	.0150	....	.0152	....
.0144	....	.0142	....	.0142	....	.0125	....
.0078	....	.0126	....	.0121	....	.0110	....
.0147	....	.0108	....	.0124	....	.0122	....
.0138	....	....	....	.0120	....	.0111	....
.0149	....	....	....	.0114	....	....	....
[1].0111	....	[1].0144	....	[1].0131	....	[1].0129	....

[1] Average.

In every case the amount of phloroglucid obtained was too small to permit calculation to pentosan. While the results just given appear to be somewhat contradictory, as it is reasonable to suppose that the hydrolysis would be additive in proportion to the increase in time, it must also be remembered that in working with such small quantities and with a method designed to determine a somewhat indefinite organic compound there are many sources of error which will affect the results. A comparison of these data with those given in Table VI shows that the hydrolysis of the pentosans is mainly due to the action of the water at the temperature employed. The data are compared in the following table, the average weights of phloroglucid being used:

TABLE VIII.—*Comparison of hydrolysis of pentosans in the presence and absence of diastase.*

Time.	With diastase, phloroglucid.	Without diastase, phloroglucid.	Hydrolysis due to diastase, phloroglucid.
	Grams.	*Grams.*	*Grams.*
One-half hour	0.0139	0.0111	0.0028
One hour	.0157	.0144	.0013
One and one-half hours	.0111	.0131	....
Two hours	.0189	.0129	.0060

The only conclusion which can be derived from these data is that the hydrolysis of the pentosans, which is mainly due to the action of the water present, is so small as to be a negligible quantity.

THE ESTIMATION OF THE PENTOSANS.

The circular letter sent out by the referee in 1898 requested that the pentosans be determined in the bran. The following results have been obtained:

TABLE IX.—*Results obtained with the phloroglucol method.*

Analyst.	Bran I.		Bran II.	
	In original material, pentosans.	Calculated to dry basis, pentosans.	In original material, pentosans.	Calculated to dry basis, pentosans.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Hatch Experiment Station	17.30	19.27	16.52	18.36
G. S. Fraps, North Carolina College of Agriculture	18.10	20.43	16.05	18.44
C. P. Beistle, Pennsylvania State College	17.84	19.82	15.61	17.71
C. A. Browne, jr., Pennsylvania State College	....	....	15.61	17.71
W. H. Krug, United States Department of Agriculture	18.41	20.79	16.31	18.50
Averages	17.91	20.08	16.02	18.14

COMMENTS OF ANALYSTS.

E. B. Holland.—Bran No. I: The phloroglucid was dried in three periods of four hours, two hours, and three hours, respectively. At the end of the third period all showed a gain over the second weight. Used 0.85 gram phloroglucol with each test and dissolved it in hydrochloric acid at 88°. The phloroglucol used was Merck's chemically pure, still it reacted strongly for diresorcol by test. Bran No. II: Used 0.9 gram phloroglucol with each test. Dissolved it in hydrochloric acid (sp. gr. 1.06) at 80° C. Employed Merck's chemically pure phloroglucol, though it reacted strongly for diresorcol. As our burners were not sufficiently powerful with the gas pressure then available to use gauze tops and accomplish the distillation of the furfural in the desired time, the flasks were set on wire gauze surrounded by the asbestos paper, with a hole in the center, which allowed the heat to strike the bottom of the flask, but prevented it from striking the sides and charring the material.

G. S. Fraps.—It was found better to distill on a wire gauze than in a bath of Rose's metal. No trouble was experienced from charring when the 30 cc of acid were added all at once, and in such a manner as to wash down the particles adhering to the sides of the flask. I consider it best to distill off 360 cc without regard to the quantity of pentosans present. All of the substance which is precipitated with phloroglucol does not pass into the first 360 cc. In an experiment on bran No. 2, 0.004 gram of phloroglucol was precipitated from a second 360 cc, equivalent to 0.13 per cent pentosan. Drying requires from seven to nine hours in the water oven. It was found impossible to dry to constant weight in three or four hours.

Following are the various determinations made on brans No. 1 and 2, of which the figures appearing opposite my name in Table IX are the average:

Estimation of pentosans with the phloroglucol method.

Determinations.	Bran No. 1.		Bran No. 2.	
	Pentosans in original material.	Per cent calculated to dry basis.	Pentosans in original material.	Per cent calculated to dry basis.
1	18.20	20.54	16.01	18.40
2	17.98	20.29	16.19	18.61
3	18.14	20.47	16.05	18.49
4	....	....	16.07	18.47
5	....	....	15.83	18.25

The phloroglucol used for Bran No. 1 was pure only on determination 2, in which Merck's phloroglucol free from diresorcol was used. The Merck's reagent was used in determinations 1 and 2 of bran No. 2. It gave no test with acetic anhydrid and sulphuric acid. Determinations 3, 4, and 5, of bran No. 2, were made with a phloroglucol purified as follows: 11 grams of phloroglucol (impure) were added slowly, with stirring, to 300 cc of hydrochloric acid (1.06 sp. gr.) heated in a beaker and stirred well until almost all dissolved. Some impurities may not dissolve. The liquid was poured into 1,200 cc of the same acid (cold) and the diresorcol allowed to crystallize out. It is filtered immediately before using. The volume containing the required amount of phloroglucol is added to the solution containing the furfural. Over 120 cc would hardly be required in any case. I have used the solution above mentioned in the determination of pentosans in a number of fodders and excrements and found it to give the same results as Merck's chemically pure. The difficulty with impure phloroglucol is that the diresorcol is difficultly soluble in the hydrochloric acid, and is left on the filter. The method of purification just described allows the larger part of the diresorcol to crystallize out, and what remains can do no harm, since it is in solution.

C. A. Browne, jr.—Distillation made from 20-ounce Erlenmeyer flasks. No charring of material upon walls of flask.

DISCUSSION OF RESULTS.

The data given in Table IX are in general very gratifying, as they demonstrate the value of the phloroglucol method and its superiority over the phenylhydrazin method. Mr. Holland reported the results obtained by him in detail and I include them at this point, as they appear to explain to some extent the variations occurring in the results.

TABLE X.—*Influence of time upon the amount of pentosans obtained by the phloroglucol method.*

Number.	Bran I.		Bran II.		
	Average time of distillation.	Pentosans in dry matter.	Average time of distillation.	Pentosans in dry matter.	Filtrate.
	Minutes.	*Per cent.*	*Minutes.*	*Per cent.*	
A	$11\frac{1}{4}$	[a]19.82	$9\frac{2}{3}$	[c]16.63	Clear.
B	$11\frac{2}{3}$	[a]19.58	$8\frac{9}{10}$	[c]16.64	Do.
C	$13\frac{1}{3}$	[b]18.82	$8\frac{4}{5}$	[d]16.46	Cloudy.
D	15	[b]18.85	$11\frac{1}{3}$	[d]16.34	Do.

[a] Distilled in the morning and stood over night.
[b] Distilled in the afternoon and stood over night.
[c] Precipitated solution, stood 20 hours.
[d] Precipitated solution, stood 15 hours.

These figures bring out an important point, namely, that the method should be more specific as to the time the precipitated distillate must stand before the phloroglucol is filtered off. They indicate that the time should be at least 20 hours, and it is quite probable that the differences found in Table IX are partly due to variations in the time which intervened between the addition of the phloroglucol and the final filtration.

Mr. Fraps obtained on bran No. 1 with a phloroglucol which contained diresorcol 20.54 per cent, 20.47 per cent, and 20.43 per cent of pentosans calculated to dry matter, while with a chemically pure product he obtained 20.29 per cent. On bran No. 2 he obtained with Merck's chemically pure phloroglucol 18.40 per cent and 18.61 per cent of pentosans calculated to dry matter, while with a phloroglucol purified by himself he obtained 18.49, 18.47, and 18.35 per cent.

It appears to be extremely difficult to obtain a phloroglucol quite free from diresorcol in the market, the absolutely pure product being quite expensive. I therefore think that the method of purification employed by Mr. Fraps is worthy of adoption. Mr. Fraps has lately published a paper on the purification of phloroglucol,[1] in which he shows that the solution of phloroglucol as prepared by him gives as good results as Merck's product free from diresorcol. On three solutions of furfural the weights of phloroglucol were:

Comparison of phloroglucol purified according to Fraps with Merck's phloroglucol.

	I.	II.	III.
Purified phloroglucol	0.4575	0.5115	0.4647
Merck's, free from diresorcol	0.4620	0.5124	0.4671

The distillate obtained by the phloroglucol method very often contains small quantities of solid fatty acids, which necessarily are a source of error, as they increase the weight of the precipitated phloroglucol. In Bulletin No. 172 of the North Carolina College of Agriculture and Mechanic Arts Mr. Fraps describes the method as used by him and states that he removes this source of error by allowing the distillate to pass through a small filter paper. This is without doubt a valuable improvement.

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[1] American Chemical Journal, Vol. XXIV, No. 3, p. 270.

THE ESTIMATION OF GALACTAN.

Very little work has been done during the last two years on the method provisionally adopted in 1898, and the results are so meager that it is not possible to safely draw any deductions from them. They are given in the following table:

TABLE XI.—*Results obtained with the Galactan method.*

Analyst.	Peas.		Clover seed.	
	In original material, Galactan.	Calculated to dry basis, Galactan.	In original material, Galactan.	Calculated to dry basis, Galactan.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Hatch Experiment Station	0.78	0.88	4.47	4.81
C. A. Browne, jr., Pennsylvania State College			4.43	4.78
O. W. Knight, Maine Experiment Station	0.48	0.54		
W. H. Krug, United States Department of Agriculture			4.29	4.79
Averages	0.63	0.71	4.40	4.79

COMMENTS.

E. B. Holland.—Peas: Mucic acid collected in a weighed filter and dried three hours. Clover seed: After heating with ammonium carbonate the macerated filter paper should be washed several times with hot water by decantation. Upon treating with nitric acid the last time, allow it to stand one-half hour or longer, as the bleaching action of the acid, if continued, will often yield a whiter, cleaner product. I should advise, by all means, the use of the tared filter on which to collect the mucic acid, as the limited quantity of wash liquids has much greater action on the precipitate when collected in the small point of the filter than when spread out on the broader surface of the Gooch crucible. The filters should be weighed in every case in light glass-stoppered weighing vessels.

RECOMMENDATIONS.

Phloroglucol method.—It is recommended that—

(*a*) The fifth line be changed so as to read "regulated so as to distil 30 cc in about ten minutes, the distillate passing through a small filter paper."

(*b*) In the sixth line, after "dilute acid," the following words be added: "added in such a manner as to wash down the particles adhering to the sides of the flask."

(*c*) The distillation be continued until the distillate amounts to 360 cc.

(*d*) In the eighth line the words "free from diresorcol" be changed to "purified if necessary."

(*e*) In the fourteenth line the words "over night" be changed to "at least twenty hours."

(*f*) The method of purifying the phloroglucol devised by Mr. Fraps be adopted and added to the paragraph entitled "qualitative test of the purity of the phloroglucol." The method is as follows:

About 300 cc of hydrochloric acid, 1.06 sp. gr., are heated in a beaker and 11 grams commercial phloroglucol added in small quantities at a time, stirring constantly until it has almost entirely dissolved. Some impurities may resist solution, but it is unnecessary to dissolve them. The hot solution is poured into a sufficient quantity of the same hydrochloric acid (cold) to make the volume 1,500 cc. It is allowed to stand at least over night—better several days—to allow the diresorcol to crystallize out, and filtered immediately before using. The solution may turn yellow, but this does not interfere with its usefulness. In using it, the volume containing the required amount is added to the distillate from the pentosan.

Galactan method.—It is recommended that in the fourteenth line, after "ammonia," the following be added: "The filter paper and contents are then washed several times with hot water by decantation, the washings being passed through a filter paper, to which the material is finally transferred and thoroughly washed."

It is finally recommended that the terms "phloroglucol," "diresorcol," and "furfural" be adopted.

SUGGESTIONS.

It is suggested that—

(*a*) The present method of determining the moisture in fodders be further investigated, with a view to fixing the time required and the exact temperature at which the determination must be made.

(*b*) The effect of various methods of distillation on the results obtained by the phloroglucol method be investigated.

(*c*) The effect of the length of time which the precipitated distillate stands on the amount of phloroglucol obtained be determined.

NOTES ON THE ESTIMATION OF SUGARS BY WEIGHING THE PRECIPITATED CUPROUS OXID AS SUCH AND AS METALLIC COPPER.[1]

By O. W. Knight.

In the estimation of sugars it has been the almost universal practice of chemists to reduce the precipitated cuprous oxid to metallic copper before weighing it, with a view of ascertaining whether equally accurate results could not be obtained by weighing the cuprous oxid and calculating it to metallic copper, the following experiment was undertaken:

A fixed amount of Fehling solution was acted on by different amounts of invert sugar in solutions of equal bulk, and the precipitated oxid filtered into a Gooch crucible washed with water and alcohol, dried at 100° to a constant weight, which took about one hour, and weighed. The weight of copper was calculated therefrom. The cuprous oxid was then dissolved in dilute nitric acid filtered into a platinum evaporating dish, the nitric acid evaporated off and replaced by sulphuric acid, and the metallic copper determined by electrolysis. A table showing the comparison of results by these two methods is appended.

Results obtained by weighing the cuprous oxid as such, and as metallic copper.

Solution.	Method No. 1.		Method No. 2.	Difference.
	Weight of cuprous oxid.	Weight of copper as calculated.	Weight of copper as determined	
	Grams.	*Grams.*	*Grams.*	*Grams.*
1	.2640	.2344	.2340	.0004
2	.2586	.2296	.2294	.0002
3	.2504	.2224	.2224	.0000
4	.3290	.2922	.2922	.0000
5	.3298	.2929	.2929	.0000
6	.3850	.3419	.3380	.0039
7	.3786	.3362	.3370	—.0008

At this point in the proceedings Mr. Wheeler exhibited to the members of the association a special form of extraction apparatus.

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[1] Presented too late to be read at the meeting.

Mr. Fraps then presented the following paper:

NOTES ON THE DETERMINATION OF PENTOSANS AND CRUDE FIBER.

By G. S. Fraps.

During the past year the writer has done considerable work upon pentosans, and has adopted some modifications of the provisional method of the association that seemed desirable. Some of these modifications have been communicated to the referee, and have been included in his report, but it has seemed best to make them, together with some other points connected with the determination of pentosans and crude fiber, the subject of a further communication. With regard to the pentosans there are several points to be taken up, the first being the method of distillation.

THE METHOD OF DISTILLATION.

In determining pentosans the material is distilled with 100 cc of hydrochloric acid of 1.06 sp. gr., and, as soon as 30 cc has distilled, 30 cc of fresh acid are added. The association does not prescribe the kind of flask to be used for the distillation, or the manner in which the fresh addition of acid is to be made. I have seen inexperienced analysts make the addition of acid by taking the stopper out of the flask and pouring it in. While the statement may not be necessary for advanced workers, it would be safer to require the acid to be added through a separatory funnel.

Whether the acid is added slowly or all at once is also of some importance. The ebullition of the liquid throws part of the material up on the sides of the flask, especially when it is rich in crude fiber, where it is to some extent withdrawn from the action of the acid, and is also in danger of being charred. If the acid is added in such a manner as to wash these particles down into the liquid, the quantity washed up again during each interval will be less and less, until this precaution is no longer necessary. The acid must be added all at once, or this object can not be accomplished.

THE QUALITY OF THE PHLOROGLUCOL.

It is safe to say that if a phloroglucol free from diresorcol (costing about $16 per ounce) must be used for the determination of pentosans, very few determinations will be made by the chemists of this country. The writer has very good reason to believe that the method of purification devised at the North Carolina Station, and recommended by the referee for adoption, will be found satisfactory. The theory of this method of purification is very simple. The principal impurity of commercial phloroglucol is diresorcol, which is insoluble in hydrochloric acid of 1.06 sp. gr., and is accordingly precipitated with the phloroglucid precipitate. The method of purification is to dissolve the phloroglucol in hot acid of this strength; part of the diresorcol does not dissolve, part of that in solution crystallizes out on cooling. The solution of phloroglucol in hydrochloric acid will keep for some weeks, but not indefinitely. If kept too long the results will be too high. A solution preserved in the laboratory for about eight months gave results about 0.2 per cent too high.

PRODUCTS OF THE DISTILLATION OF PENTOSANS WITH HYDROCHLORIC ACID.

It is assumed that when materials containing pentosans are distilled with hydrochloric acid of the proper density, the only product which is afterwards precipitated with phloroglucol is furfural. This, however, is not the case, as the following experiments show:

Freshly distilled furfural was dissolved in hydrochloric acid of 1.06 sp. gr., three portions taken, and one distilled in the usual way, until the volume of the distillate was 270 cc. The distillate was made up to 500 cc, and precipitated with phloroglucol as usual. The undistilled portions were precipitated in the same way. The results were as follows:

Precipitates from distilled and undistilled portions of fresh furfural.

Phloroglucid.	Weight of precipitates.		Mean.
	Gram.	*Gram.*	*Gram.*
Undistilled portion	0.2815	0.2805	0.2810
Distilled portion	0.2804	....	0.2804
Loss	....	....	0.0006

Furfural, therefore, suffers no appreciable loss by distillation with hydrochloric acid. The solution of furfural in hydrochloric acid became brown after standing four days, probably by oxidation. Five portions of the brown solution were taken, and three distilled as just described. The results of these experiments were as follows:

Precipitates from distilled and undistilled portions of furfural after standing.

Phloroglucid.	Weight of precipitates.			Mean.
	Gram.	*Gram.*	*Gram.*	*Gram.*
Undistilled portions	0.2880	0.2863	....	0.2871
Distilled portions	0.2785	0.2779	0.2762	0.2775
Loss	....	....	....	0.0096

When the furfural is exposed to the air for four days and distilled with hydrochloric acid it loses 3.2 per cent in weight.

Now, when vegetable materials were distilled with hydrochloric acid in the usual way for the determination of pentosans, and the distillates divided into two portions and one portion distilled as described above, and the other precipitated directly, the dfferences were always found to be greater than 3.2 per cent. Some body, which, unlike furfural, is easily affected by hydrochloric acid, must be present in these distillates. The quantity of this body is different for different materials, as may be seen from the following tables:

Distillates stood over night before precipitation or redistillation.

Materials.	Loss.
	Per cent.
Cotton-seed meal	7.4
Corn silage and corn bran	15.6
Cowpea meal	17.9
Crimson clover hay	11.7
Irish potato vines	10.2
Corn bran	22.6

Distillates precipitated or distilled the same day.

Materials.	Loss.
	Per cent.
Pine straw	13.2
Oxycellulose	13.8
Gum arabic	12.9
Corn cobs	10.1
Okra hulls	11.5
Oak sawdust	6.9

These figures go to show that when vegetable materials are distilled with hydrochloric acid there is formed, besides furfural, one or more bodies which are easily changed by the hydrochloric acid. This investigation is still in progress, and will be pushed to completion when possible.

Before passing on, it should be noted that when the distillate stands for a time usually a black precipitate settles out in small quantity. This was always filtered out before working on the solution.

NOMENCLATURE.

Some of the terms used by the association in the description of the pentosan determination are not in accord with the best terminology. The ending "ol" has been adopted generally to designate the alcoholic or phenolic character of a body. For this reason the term furfurol is objectionable, since the body in question is not an alcohol or a phenol but an aldehyde. Either "furfural" or "furfuraldehyde" would be better. Both are in use. The substances which are called phloroglucin and diresorcin are phenolic in character and should terminate in "ol." Making the same change which converts glycerin to glycerol, we change these names to phloroglucol and diresorcol.

The term pentosan is not fully justified in the light of our present knowledge. We assume that all the furfural obtained by distilling vegetable materials with hydrochloric acid is derived from pentosans, meaning thereby a body $(C_5H_8O_4)_x$ which would be analogous to the hexosans starch $(C_6H_{10}O_5)_x$ or cellulose $(C_6H_{10}O_5)_x$. But it is well known that oxyhexoses will yield pentosans under the same conditions, examples being glycuronic acid $(COH.\ (CHOH)_4\ COOH)$, osones, $(CHOH(CHOH)_3\ CO.\ COH)$, and oxycelluloses, which is cellulose in which one or more of the hexose groups has been oxidized to groups like glycuronic acid or osones.

Cross and Bevan propose to call the furfural-yielding substances furfuroids (furfuran-like). A better term, it seems to me, would be pentosoid—meaning pentose-like—and these bodies are like pentoses in that they yield furfural.

CRUDE FIBER.

When one attempts to introduce the determination of pentosans into his scheme of analysis for feeding stuffs he meets with difficulty, in that crude fiber usually contains pentosans. Crude fiber may contain 40 per cent of the pentosans present, and often contains 20 per cent. For example, 14.4 per cent of the crude fiber of timothy hay is pentosans, and 20.5 per cent of the pentosans are contained in the crude fiber; 13.4 per cent of the crude fiber of crab-grass hay is composed of pentosans, and 19.6 per cent of the pentosans are in the crude fiber. Evidently pentosan can not be subtracted from nitrogen-free extract, and the only way to place this determination in a systematic analysis of feeding stuffs is to determine it in the crude fiber also, that is if we retain our present method for crude fiber. The pentosans in the crude fiber are different from the pentosans in the nitrogen-free extract, for they are digested to a less extent than the latter or the crude fiber. In spite of this such a separation is entirely arbitrary, and it would be better to adopt a method for the crude fiber determination in which the pentosans are dissolved completely. Such a method has been devised by J. König [Analyst, **23**, 47 (abstract)], and is as follows:

Three grams air-dried substance are placed in a porcelain basin with 200 cc glycerol of 1.23 sp. gr., containing 20 grams concentrated sulphuric acid per liter. The basin is placed in an autoclave and steamed for 1 hour at 137° C. It is allowed to cool to 80°–100° C, 200–250 cc water added and the contents filtered while hot, washed, and ignited as usual. In the absence of an autoclave, the digestion may be carried out in a 600 cc flask connected with a reflux condenser. The glycerol is brought to a very gentle boil, boiled 1 hour, and finished as before.

The crude fiber prepared by this method is practically free from pentosans. Kellner, Herring, and Zähn[1] conclude from their study of this method that it is an improvement over the laborious method now in use, and recommend it for use in digestion and metabolism experiments as being more accurate, more rapid, and more easily carried out than the present method, and as effecting a sharper separation of constituents.

Mr. Wheeler presented the following resolution and requested that the same be transmitted as soon as possible, so that it might be read before adjournment:

Resolved, That the thanks of the association be extended to the National Grange Patrons of Husbandry for the several courtesies extended to this association.

Motion carried.

The president then called for the report on dairy products, and the referee being absent the report was read by Mr. Frear, as follows:

REPORT ON DAIRY PRODUCTS.

By J. B. WEEMS, *Referee,* and F. W. WOLL, *Associate Referee.*

The time for the investigation in connection with the work of the association being so short during 1899, it was decided to continue the work of last year and in addition to investigate other methods for the determination of casein and albumin, of interest in this connection.

In preparation for the work circular letters were sent to all chemists connected with experiment stations, asking their cooperation in the work of the year. The result of these requests was that there were received twelve favorable replies, and to each of the chemists offering to aid in the work a sample of skim milk preserved with formaldehyde was sent with the following circular:

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

METHOD OF ANALYSIS OF DAIRY PRODUCTS.

Your cooperation is respectfully requested in a study of the following methods for the determination of casein and albumin in milk:

I. (*a*) *Casein, official method:*

Determine the amount of casein in the sample of milk as given under "Official method of determination of casein in cow's milk," p. 55, par. 1, Bul. 46, rev. edition, Division of Chemistry, Dept. of Agriculture.

(*b*) *Albumin, provisional method:*

Determine the albumin in the filtrate of the above, as outlined in "Provisional method of the determination of albumin in milk," p. 55, par. 2, Bul. 46, rev. edition.

II. (*a*) *Casein, official optional method:*

Determine the casein as given in the "Official optional method for determining casein in milk," p. 55, par. 3, Bul. 46, rev. edition.

(*b*) *Albumin, provisional optional method.*

Determine albumin in the filtrate of the above method as given in par. 4, "Provisional optional method for determining albumin in milk," p. 55, Bul. 46, rev. edition.

III. (*a*) *Casein, modification of official method:*

Carry the method through as described, to and including the washing of the precipitate, then neutralize filtrate as nearly as possible with sodium hydroxid, filter off any precipitate that may form and add it to the former precipitate for the nitrogen determination.

(*b*) *Albumin, modification of provisional method:*

To the filtrate from the preceding determination add 10 cc of 20 per cent tannin solution; heat, filter, wash with water, and determine nitrogen.

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[1] Exp. Station Record (abstract), XI, 705.

IV. (*a*) *Casein, alum method:*

To 10 cc of milk add 50 cc of water at 40°. Then add 1.5 cc of alum solution saturated at 40° or higher. If the precipitate fails to settle promptly add 0.5 cc more of the alum solution; filter, wash with water, and determine the nitrogen.

(*b*) *Albumin, modification of provisional method:*

To the filtrate from the preceding determination add 10 cc of 20 per cent tannin solution; heat, filter, wash with water, and determine nitrogen in the usual manner.

V. *Indirect, or Penny's method:*

(*a*) Dilute 25 cc of milk with 150 cc of distilled water in a 250 cc measuring flask. Then add 5 cc of alum solution (see above, IV (*a*)) to precipitate the casein, and fill to the mark. Determine the nitrogen in 100 cc of the filtrate in the usual manner.

(*b*) Dilute 25 cc of milk with 150 cc of distilled water in a 250 cc measuring flask. Then add 25 cc of 20 per cent tannin solution to precipitate the casein and albumin; heat, cool, fill the flask to the mark with the distilled water and filter. Determine the amount of nitrogen in 100 cc of the filtrate.

(*c*) Determine total nitrogen in the sample of milk by the Kjeldahl method. Multiply the amount of nitrogen found in *a*, *b*, and *c* by 6.25, and from these results calculate the percentages of casein and albumin in the sample of milk.

In reporting results give separate figures for *a*, *b*, and *c*.

In reporting the analytical results for the above methods please give all the data and information that will be of interest. We shall be glad to have you state which methods you regard most favorably, and the considerations or reasons for your opinion, as well as any objections which you may find to any of the methods.

Yours, very respectfully,

J. B. Weems, *Referee.*
F. W. Woll, *Associate Referee.*

The results obtained by the chemists cooperating in the work were as follows:

Results of the comparison of methods for the determination of casein and albumin in milk, 1900.

Analyst.	Casein.					Albumin.					Total nitrogen × 6.25.
	I.	II.	III.	IV.	V.	I.	II.	III.	IV.	V.	
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Department of feeds and feeding, Massachusetts Experiment Station	2.76	[1]3.02	2.78	2.69	2.67	0.18	0.31	0.51	0.59	[1]0.02	3.42
C. B. Cady, Missouri	2.69	2.88	2.84	2.65	2.65	.32	.28	......	.60	.61	3.31
H. A. Huston and A. H. Bryan, Indiana	2.78	2.83	2.85	2.83	2.81	.27	.45	.48	.50	.46	3.39
John White and R. W. Thatcher, Nebraska	2.80	2.67	2.80	2.65	2.54	.14	.10	.31	.49	.59	3.26
C. L. Penny, Delaware	2.68	......	2.77	2.68	2.68	.18	......	.35	.49	......	3.30
J. A. LeClerc, Geneva, N. Y.	2.71	2.84	2.72	2.71	2.69	.30	.32	.44	.53	.49	3.35
T. M. Price, Maryland	2.42	2.71	2.63	[1]2.32	2.45	.38	.43	.53	.44	.53	3.15
F. W. Woll and Alfred Vivian, Wisconsin	2.67	2.68	2.66	2.71	2.48	.18	.29	.36	.35	.59	3.27
C. E. Ellis, Iowa	2.65	2.74	2.72	2.64	2.64	.22	.27	.47	.44	.58	3.22
C. E. Gray, Iowa	2.74	2.70	2.75	2.71	2.54	.31	.30	.48	.61	.54	3.27
Average	2.690	2.756	2.752	2.697	2.615	.248	.306	.437	.504	.549	3.294
Highest	2.80	3.02	2.85	2.83	2.81	.38	.45	.53	.61	.61	3.42
Lowest	2.42	2.67	2.63	2.32	2.45	.14	.10	.31	.35	.02	3.15

[1] Omitted in average.

COMMENTS OF THE ANALYSTS.

The department of feeds and feeding of the Massachusetts Experiment Station makes the following remarks upon the work:

> The official optional method for casein proved objectionable because of the slow filtration and difficult washing of the precipitate, due to the crystallizing out of the magnesium sulphate on the filter as the solution cooled.
>
> In the alum method undoubtedly the addition of 2 cc of alum would insure perfect precipitation and a clear filtrate, which was not obtained in every case with 1.5 cc.
>
> In the direct method 5 cc of alum did not yield a clear filtrate and the filtration was exceedingly slow. Again, in the indirect method 25 cc of tannin solution apparently did not precipitate all the casein and albumin, judging from the results, though the filtrate was clear.
>
> After considering both the manipulation of the several methods and the results obtained thereby we should be inclined to indorse the modified official method for casein without a second filtration after neutralizing the soda, and the modified provisional method for albumin.

Mr. Cady's experience with the methods is shown in his conclusions:

> In "Section III, Modification of Official Method," after neutralizing the filtrate from the acetic-acid precipitate with sodium hydrate, I obtained a somewhat gelatinous precipitate, which filtered with much difficulty, giving very little albumin when treated as directed.
>
> The tannic acid which I used was marked C. P. However, on making blank determinations, using 10 cc of the 20 per cent solution, I found that 10 cc of the solution contained 0.0054 gm of nitrogen, and acting on the supposition that all of the nitrogen contained in the tannin solution was retained in the filtrate, I made reduction accordingly.

Mr. H. A. Huston drew conclusions as follows regarding the methods:

> The magnesium-sulphate method has always proved very tedious in our hands, while the method of Penny has the merit of avoiding long filtrations and washings, although requiring two extra nitrogen determinations.

Mr. John White reports as follows regarding the methods:

> The official method for casein appears to be the most satisfactory one. It is rapid, easy of manipulation, and gives very concordant results. The suggested modification of this method, viz, the neutralization of the filtrate by sodium-hydrate solution and refiltration, appears to be of little value, since no appreciable precipitate is formed on neutralization.
>
> The alum method is also a favorable one for casein. It gives somewhat lower results than the precipitation by acetic acid, but otherwise is quite satisfactory.
>
> The official optional method does not appear to be satisfactory. It would seem from observations made that the proportion of nitrogenous bodies precipitated by magnesium sulphate varies between limits too wide to justify the use of this precipitant. Further, the use of a substance which crystallizes so readily as does magnesium sulphate in a solution saturated at a temperature above the working temperature of the laboratory causes obvious difficuties in manipulation which are hard to overcome.
>
> Concerning the methods for the precipitation of albumin, tannic acid seems to be by far the most satisfactory precipitant. It yields somewhat higher results than does acetic acid, but the results are very concordant, the differences in the percentages obtained from the different filtrates being apparently due to differences in the proportion of nitrogenous bodies precipitated as casein by the various precipitants used.
>
> The indirect or "Penny's" method is rather a neat one and easy of manipulation. It does not, however, appear to possess any particular advantage over the direct method and is not quite so rapid, more nitrogen determinations being required.

Mr. LeClerc gives the results of his experience in the work in the following comments:

> *Method II* (*a*).—Required six hours to filter and wash. Magnesium sulphate will precipitate any globulin that may be in milk. The washing takes so long that possibly the precipitate is not absolutely free from albumin. Hence a slightly higher result for casein.
>
> *Method III* (*a*).—I was unable to obtain a precipitate on neutralization. Got only a slight cloudiness, which was not removed even after repeated filterings.
>
> *Method IV* (*a*).—Obtained good precipitations, but found it necessary to have the solution, after adding the water, at least 40° C. Thorough washing of the precipitate

to remove all traces of alum is necessary, because the alum sometimes contains ammonium as impurity.

Method V (*a*).—This method works very well, provided precipitation is done at about 42° C. and care is taken that the alum is not a mixture of potash and ammonium alum. I had much difficulty with this method till I found that my alum contained an ammonium salt.

I prefer Methods I (*a*) and IV (*a*).

It has been shown in this laboratory by Van Slyke and Hart that milk generally contains nitrogen compounds other than casein and albumin, and that these bodies are thrown down by tannin. Hence higher results in all cases where tannin was used as a precipitate for albumin.

Mr. C. L. Penny makes the following comments on the results obtained in carrying on the line of work outlined by the referee:

I (*a*).—I find acetic acid a good precipitant and convenient to use.

(*b*).—Filtration slow in many cases, but for the most part the method worked well in the execution. The results, 0.183 per cent, were far too low, being about 30 per cent of the true result.

II (*a*) and (*b*).—The filtration was exceedingly slow, so slow, in fact, that this method was abandoned; hence I have no analytical results. My impression is that Dr. Van Slyke had a similar experience with the magnesium-sulphate precipitation. I can not find the reference now; it was probably a private communication. At all events, magnesium sulphate did not seem to promise enough to justify the expenditure of more time.

III (*a*).—The neutralization of the filtrate, contrary to my former experience as reported, does give an additional precipitate, amounting in this case to about one-tenth of 1 per cent, reckoned on the original sample. The second filtration I found in one case fairly rapid, in two other cases very slow, so slow that I abandoned that part of the work. I think a double filtration is hardly practicable.

(*b*).—The tannin filtration was fairly rapid, but not very clear. The result was far too low, but little over half of the true one.

IV (*a*).—Alum seems to me to be the best precipitant. The precipitate is the easiest and quickest to wash. The several results are very concordant. I think that alum furnishes as good a practical reagent for separating albumin from casein as any that I know of.

(*b*).—The tannin here gave a good precipitate and a clear filtrate. The filtration was rapid. The result was nearer the truth for albumin than that given by any other precipitant. The several results are also concordant.

V (*a*).—There is little more to be said about alum than has been said above, so far as my experience goes. The precipitation was good, the filtration rapid, and the filtrate clear. I evaporated 100 cc in Kjeldahl flasks after adding 30 cc of strong sulphuric acid over a slow flame, in some cases with an air blast and in some cases without. I would prefer 50 cc (representing 5 cc of milk) to 100 cc on account of the time spent in reducing the volume of the solution. An air blast facilitates this part of the operation, however, greatly. With 50 cc of filtrate the solution in the Kjeldahl flask may be brought down, by careful adjustment of the flame, to a point where there is no danger of foaming. Thus I prefer 50 cc to 100 cc, and I believe it will be quite accurate enough, for the nitrogen determinations in milk agree much more closely in my experience than those of most other bodies.

(*b*).—The filtrate from tannin in my work showed 0.044 per cent of nitrogen or 0.277 per cent of protein. But the tannin solution itself contained, in the quantity used to precipitate nitrogen equivalent to 0.225 per cent of protein, i. e., had the milk sample been free from all nitrogen, the tannin alone would have shown 0.225 per cent of protein. Hence this test falls out as entirely worthless, as I have made it, and I therefore omit its results from the table, as it shows absolutely nothing.

(*c*).—The total nitrogen was determined in 10 cc of milk directly in the usual way by the Kjeldahl method.

Supplementary.—I precipitated 25 cc of milk in the 250 cc flask with acetic acid (10 per cent), 4 cc as nearly as possible according to the official method for casein, I (*a*), so far as dilution and temperature were concerned; but necessarily the dilution was somewhat less, viz, 150 cc of water, or sevenfold instead of tenfold, as in the official method, for casein. The filtration was speedy and the filtrate clear; the precipitant seemed to be an excellent one. The result was a trifle lower than by V (*a*), with alum, viz, 0.58 per cent instead of 0.62 per cent with the latter. As between alum and acetic acid, I have but little preference; if any, it is for alum. Perhaps, however, the alum is the more uniform and the acetic acid the more capricious, the latter depending more on details of treatment than the former.

In conclusion, as to a final choice of methods, I like the method of difference for the following reasons:

(1) The filtration is exceedingly rapid and there is no washing of the precipitate.

(2) The total protein is sure to be given accurately by this method, and that, in fairness to the sample, is most important. I fear a shortage in albumin if it be determined by actual precipitation with heat or tannin.

(3) The difference method uses but a single reagent to precipitate, either alum or acetic acid as may be decided on.

(4) Of the three quantities, total protein, casein, and albumin (the first of which is practically, if not absolutely, equal to the other two added together), the method of difference determines in any event the one that is by far the easiest, viz, total protein, instead of determining the more difficult two, casein and albumin.

Mr. T. M. Price reports as follows:

I prefer the alum method, as the manipulation is such that it precipitates rapidly and filters very easily, but I find that the alum method seems to give a little lower results than the others. The regular official method is very easy to work and gives no particular trouble. In the optional method the filtration is very slow, and we sometimes had difficulty because of the crystallizing of the magnesium sulphate in the stem of the funnel.

Mr. F. W. Woll makes the following comments:

As to the comparative value of the different methods I may say that my preference is decidedly the alum method or the Penny method, if either is found correct. The precipitated casein in the former method comes down in large clots that allow of rapid filtration. If total nitrogen, casein, and albumin are to be determined in a sample of milk, the Penny method would be very convenient, provided we can depend on the results. In Method III no precipitate of casein was obtained by neutralizing the filtrate with soda solution, but you will notice that the albumin was increased. The per cent of casein in Methods I and II ought, therefore, to have been the same.

In comparing the results obtained from the sample of milk it will be noticed that the average for Method I, or the official method, was 2.69 per cent, and for Method IV, or the alum method, was 2.697 per cent, a difference of 0.007 per cent. After considering these results and the reports by the analysts, as well as the results obtained in previous years, the reporters feel warranted in making the following recommendation:

(1) That paragraph 3 of Bulletin 46, revised edition, p. 55, entitled "Official optional method for determining casein in milk," "To 5 grams of milk, 50 cc of a solution of magnesium sulphate," etc., to be changed to read as follows:

(3) "*Official optional method for determining casein.* To 10 cc of milk add 50 cc of distilled water at 40°. Then add 2 cc of alum solution saturated at 40° or higher. Allow precipitate to settle, transfer to a filter, and wash. The washed precipitate and filter paper are digested as in the regular Kjeldahl method as outlined in the official method, paragraph 1. Calculate casein by multiplying the amount of nitrogen found, by 6.25.

(2) That paragraph 4 of Bulletin 46, p. 55, entitled "Provisional optional method for determining albumin milk," etc., be changed to read as follows:

"*Provisional optional method for determining albumin in milk.*"—To the filtrate from the preceding add 10 cc of 20 per cent tannin solution, heat, filter, wash with distilled water, and determine nitrogen by the Kjeldahl method. Multiply the result by 6.25 for albumin.

Mr. Ewell. I have a brief note in reference to one of the methods which comes under this head.

REPORT ON THE DETERMINATION OF THE SPECIFIC GRAVITY OF FATS AND OILS AT THE TEMPERATURE OF BOILING WATER.

By E. E. Ewell.

The temperature of boiling water has been a reference temperature much favored by chemists for this determination, because it makes possible a direct comparison of the density of practically all fats, oils, waxes, and free fatty acids of commercial importance, whether they be liquid or solid, at ordinary temperatures. The official methods of this association have included directions for this determination for several years. In accordance with these directions, the weight of a given volume of fat at the temperature of boiling water is compared with the weight of the same volume of water at the same temperature. Two methods are official for the standardization of the flasks:

(1) The determination of the weight of water contained in the flask, at a temperature of boiling water, by actually filling it at that temperature, cooling to room temperature and weighing.

(2) The determination of the weight of water contained in the flask at any convenient temperature and the use of this result in the calculation of the weight of water held by the flask at 100° C., using for this purpose the recorded data for the coefficients for the expansion of water and glass.

Inasmuch as the directions given in the official methods for this calculation contain an error and the formula given is incomplete, in that it leads only to the volume of water contained in the flask at the desired temperature instead of its weight, I was directed by the Chief of the Division of Chemistry to revise the formula and to inquire into the relative accuracy of the two methods of standardizing the flasks to be used for this determination.

The proper formula was readily arrived at and is as follows:

$$W^T = W^t \frac{d^T}{d^t}[1 + \gamma\ (T - t)], \text{ wherein:}$$

W^t = weight of water at temperature of observation;
d^t = density of water at temperature of observation;
t = temperature of observation;
W^T = weight of water at the desired temperature;
d^T = density of water at the desired temperature;
T = the temperature for which the weight is to be calculated;
γ = coefficient of cubical expansion of glass.
If T = 100°; and
γ = 0.000028; the formula becomes:

$$W^T = W^t \frac{0.95863}{d^t}[1 + 0.000028 (100 - t)].$$

The use of a formula of this kind necessarily involves sources of error of appreciable magnitude. This and certain other considerations named below made it desirable to determine experimentally the variations existing between the results of the two methods in use for standardizing flasks for this determination, and to critically examine the sources of error attending each of them.

The factors tending to produce variations between the results of the two methods may be briefly enumerated as follows:

(1) *The coefficient of cubical expansion of water*, which may be regarded as one of the best determined constants of nature. The values for the density of water at various temperatures, as given in *Landolt & Börnstein's Tabellen*, may be regarded as accurate beyond the requirements of the determination under discussion.

(2) *The coefficient of the cubical expansion of glass*, which is a matter of some doubt. It varies with the chemical composition of different kinds of glass and with the physical changes to which a given kind of glass may be subjected. A value for this coefficient commonly found in text-books is 0.000026. The recorded results for different kinds of glass vary from 0.000017 to 0.0000305. Schulze[1] in the course of his

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[1] Alfred Schulze. Ueber die Ausdehnung der wichtigsten Titrirflüssigkeiten durch die Wärme. Zeit. anal. Chem., 1882, **21**, 167–177.

experiments on the coefficients of expansion of certain volumetric reagents, found that the coefficient of cubical expansion of the glass of the apparatus in use by him at that time varied from occasional values as low as 0.0000288 to 0.0000305, the most of his results lying between the number last named and 0.0000295. In the second formula which I have tentatively suggested above, and which was used in the calculation of the results that are to follow, I have inserted as the value of this constant in the general formula the number 0.000028 which is a mean value. These numbers have so many ciphers between the significant figures and the decimal point that they seem at the first glance to be of little importance. Move the decimal points two places to the right and we have the expansion of a 100 cc flask stated in fractions of a cubic centimeter for each degree of increase in temperature. Move the decimal points two more places to the right and we have the expression for the increase in capacity of a 100 cc flask when warmed from 0° to 100° C.; 0.170 and 0.305 cc. These numbers are about one and one-fourth times as great as the expansion which has to be corrected for in standardizing a 100 cc specific gravity flask for use at 100° by calculation from the results of weighings made of the water contained in the flask at ordinary temperature. The highest and lowest numbers differ by 0.135 cc, which is approximately equal to the same fraction of a gram. Such an error in the standardization of a flask would entail an error of at least one unit in the third decimal place of the result of a specific gravity determination. With proper apparatus and manipulations the error should very rarely or never pass to the left of the fifth decimal place of the result. I trust that this discussion sets forth clearly the necessity of having our specific gravity flasks made of glass of which the coefficient of cubical expansion is known with precision to the sixth decimal place at least, if we are going to standardize them for use at high temperatures by calculations based on experimental results obtained at low temperatures.

(3) *Errors attending the determination of the temperatures of observation:*

(*a*) At the temperature of the laboratory this may readily be done by means of a suitable thermometer to within 0.1°. Only a verified thermometer should be used for this work. Working at substantially the temperature of the air, the correction to be applied to the reading of the thermometers for the unimmersed portion of the column of mercury is practically zero. One-tenth of a degree of temperature means at 25° a change in the density of water of 0.000025 and approximately the same error in the result of the determination of the specific gravity of another liquid.

(*b*) The determination of the temperature of observation at or near the temperatures of boiling water involves considerably greater difficulties. If we undertake to ascertain the actual temperature by means of a thermometer, we find it necessary to apply a correction for the unimmersed portion of the mercury column of the thermometer, that may in some cases be as great as one whole degree. I doubt if this correction can be ascertained within less than 0.2 or 0.3 of a degree, involving in the latter case an error in a density determination of about two units in the fourth decimal place (0.00024). It is therefore evident that it is necessary to use a short thermometer or a form of apparatus that will permit the immersion of the thermometer to the top of the column of mercury, or very nearly to that point.

If we undertake to fix the temperature by means of boiling distilled water, we are in still greater trouble. In the first place, it is not as easy as might at first be supposed to heat water to exactly 100°, or even sufficiently near for our present purpose. We have a mass of water inclosed in a vessel whose sides are constantly radiating or conducting away heat while it is being agitated by bubbles of steam, having a temperature of the boiling point of the water or slightly more. Unless the sides of the vessel are well insulated, or are heated to or above 100° throughout their entire surface, the temperature of the water may easily be some tenths of a degree below that of the vapor it is giving off. The temperature of boiling water is, therefore, dependent upon the nature of the vessel and the vigor of the boiling. In this connection the statement made by Allen[1] is interesting:

> "It will be observed that the densities in the third column of the foregoing table are recorded as obtained at a temperature of 98° to 99° C. In the author's laboratory water ordinarily boils at 99° C., and oil immersed in a vessel of boiling water rarely reaches a temperature exceeding 98.5° C."

Now, this learned author, for whom we all have such great reverence, does not mention the influence of atmospheric pressure on the boiling point of water, and fails to state whether correction was made for the column of the thermometer not immersed in the water. In the absence of direct statements we must, of course, credit him with having made proper corrections. I have mentioned above that the correction may approach one whole degree. Allen, however, uses only 3 and 4 decimal places in stating the results given in the table mentioned in the quotation.

The matter of atmospheric pressure is to my mind the insurmountable obstacle to the use of the boiling point of water for a reference temperature for the purpose under consideration. This is certainly true when the weight of fat at the boiling point of water is compared with the weight of the same volume of water at some lower temperature, 4°, 15.5°, etc., having been proposed for this purpose. In the official method water and fat are both compared at the temperature of boiling water, and in this way the influence of variations in this temperature are eliminated in a great measure. Allen (in the table of results mentioned above) gives the specific gravities of certain oils at 15.5° C. and at 98° to 99° C., water at 15.5° C. being 1,000 in each case. Results obtained by the method of the Association of Official Agricultural Chemists can not be readily compared with results stated in the manner adopted by Allen, since the temperature of the boiling point of water may vary between comparatively wide limits. During the series of experiments reported below the barometer reading varied from 753 to 767 mm. This variation of 14 mm represents a variation in the boiling point of water of approximately 0.5° C. When we recall that at Charleston, South Carolina, and other tide-water points the mean boiling point of water is approximately 100° and sometimes more, and at Denver, Colorado, it may fall as low as 95°, the irrationality of the method is apparent. It would seem to be more scientific to direct that the specific gravities of fats and oils be determined at exactly 100°, and that the results be stated in terms of—that is, compared with—water at the same temperature. This would involve, however, the difficulty that it would be necessary to standardize the flasks by calculation in all cases in which the boiling point of water falls below 100°. For example, in high altitudes the water would begin to boil in the flasks several degrees below 100° C. It would rarely be necessary to correct by calculation for more than 5° C.

(4) *Errors attending the manipulation of filling the flask* are relatively smaller the larger the flask. These become greater as the temperature is raised, however, and are undoubtedly considerably greater at 100° than at ordinary temperatures. Evaporation is greater when the weighings of water are being made, and there is difficulty in having the stoppers at the proper temperature at the time of their insertion in the flask.

(5) *Fatigue dilatation.*—This term is used to designate a well-known phenomenon attending the heating and cooling of glass, viz, the tardiness with which a glass vessel returns to its original size when cooled to the ordinary temperature after having been heated to a higher temperature. The extent of the fatigue dilatation varies with different kinds of glass and is well illustrated by the well-known depression of the zero point of a thermometer immediately following the heating of the thermometer bulb to a high temperature.

The amount of fatigue dilatation exhibited by three specific-gravity flasks made of ordinary Bohemian glass is shown in the table of results given below. The flasks had not been used for several months previous to the experiments made on November 12 and 13. On November 15, within less than an hour after the flasks had been heated for one-half hour at the boiling point of water, weighings were made to determine the capacity of each of the flasks at room temperature. The results are given in the table, and when the calculated capacities of the flasks at 100° are compared with the mean of those calculated from the results of the four series of determinations made on November 12 and 13, it will be seen that the capacities on November 15 were 0.0022, 0.0033 and 0.0035 gram, respectively, higher than the mean of the results based on weighings made on November 12 and 13, i. e., before the flasks had been heated. These results indicate that, unless we use flasks made of glass exhibiting very small fatigue dilatation, this factor must be considered when we desire results of the highest accuracy.

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[1] Commercial Organic Analysis, Leffmann's edition, Vol. II, Pt. I, p. 31, footnote.

The error in the result of a specific-gravity determination arriving from a given error in the determination of the capacity of the flask may be readily ascertained by reference to the following table:

Error in specific gravity resulting from given error in weighing flask.

Size of specific-gravity flask.	Error in weight necessary to cause errors in specific gravity, results of—		
	0.001	0.0001	0.00001
	Gram.	*Gram.*	*Gram.*
25 cc	0.0250	0.0025	0.00025
50 cc	0.0500	0.0050	0.0005
100 cc	0.1000	0.0100	0.0010

In this connection it is proper to consider certain hypercriticisms that have been made from time to time of methods of analysis in general use. I refer to those methods which direct that a solution be prepared by boiling the substance with a solvent in a graduated flask, cooling and diluting to the mark in the same flask. It has frequently been stated by chemists, who apparently have not attempted to ascertain the amount of the attendant error, that this practice is bad; that the flask does not return to its original capacity on cooling, and consequently an error is introduced. The results cited above indicate that for glass of the quality used in the experiments reported this error for a 1-liter flask would amount, approximately, to from 0.066 to 0.088 cc. This is an error that certainly may be neglected in all ordinary routine analytical work. An estimate of the error can be arrived at in another way. The depression of the zero point of a thermometer one whole degree centigrade following the heating of the instrument to the boiling point may be considered a large depression. The fatigue dilatation is in this case 1 per cent of the apparent expansion of the mercury between 0° and 100°. For this range of temperature the coefficient of cubical expansion of mercury is 0.000182 per degree, or 0.0182 for 100°. If the coefficient of cubical expansion of glass be taken at 0.000028, the expansion of the bulb of the thermometer on being heated from 0° to 100° would be 0.0028 of its capacity at 0°. The apparent expansion of the mercury contained in it would therefore be 0.0154 of its volume, 1 per cent of which is 0.000154 of the volume of the mercury; i. e., the fatigue dilatation would be 5.5 per cent of the total expansion of the glass. If a liter flask be heated from 20° to the boiling point of water, its capacity is increased by expansion 2.24 cc. The fatigue dilatation, taken as 5.5 per cent of the total expansion, would then be 0.123 cc; i. e., the capacity of the flask would be 0.123 cc greater immediately after cooling than it was before it was heated. Even an error as great as this would be negligible in ordinary routine analytical work, but the experiments cited above and the amount of depression of the zero point exhibited by thermometers both indicate that the fatigue dilatation of laboratory glassware of ordinary quality is much less than 5.5 per cent of the total expansion it undergoes when heated. It is therefore evident that the hypercriticisms referred to above have no practical value.

I have recently completed a series of check standardizations of three flasks by the two official methods. The results are set forth in the following table:

Check standardizations of three flasks.

Date.	Temperature.	Barometer reading.	Flask No. 17.		Flask No. 77.		Flask No. 79.	
			Weight of water at T°.	Calculated weight of water at 100°.	Weight of water at T°.	Calculated weight of water at 100°.	Weight of water at T°.	Calculated weight of water at 100°.
1900.	° *C.*	*Mms.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*
November 12........	21.0	....	27.013	26.0042	51.455	49.5334	48.1915	46.3917
Do	22.7	....	27.002	26.0023	51.440	49.5355	48.175	46.3914
Do	29.3	....	26.959	26.0019	51.358	49.5337	48.098	46.3905
November 13........	36.2	....	26.903	26.0012	51.253	49.5350	47.998	46.3891
Means.........	....	....	....	26.0024	....	49.5344	....	46.3907
Maximum variations from mean..	....	....	....	0.0018	....	0.0011	....	0.0016
Maximum variations of two results.	....	....	....	.0030	....	.0021	....	.0026
	(Corrected.)							
November 14........	[1] 99.95	765	26.008	....	49.540	....	46.397	....
Do	[1] 100	764.5	....	....	49.547	....	46.388	....
Do	100	764	....	....	49.550	....	46.407	....
November 15........	100	766.8	....	....	49.540	....	[2] 46.355	....
Means.........	....	....	....	....	49.5443	....	46.3973	....
Maximum variations from means.	....	....	....	....	0.0057	....	0.0097	....
Maximum variations of two results.	....	....	....	....	.0100	....	.0190	....
Variation of means from mean calculated results	....	....	....	....	+.0099	....	+.0066	....
November 15........	21.65	....	27.010	26.0046	51.453	49.5377	48.188	46.3942

[1] These temperatures are given as accurately as they could be read with an accurate thermometer. The correction for exposed column of mercury varied from 0.8 to 0.9° C. A verified short thermometer was not available.

[2] Omitted in the calculation of averages.

RECOMMENDATIONS.

In view of these experiments and the facts set forth in the above discussion, I beg to recommend that the referee on dairy products for next year be instructed to consider the following changes in the methods for determining the specific gravity of fats and oils and present them at the next meeting for adoption by the association, together with such modifications and additions as may be found advisable in the meantime:

(1) That this determination be not made "at the temperature of boiling water," but at 100° C., using, to attain this temperature, a bath of glycerol or some other suitable substance soluble in water or miscible therewith; that the bath be so arranged that the controlling thermometer can be immersed to the 100° mark.

(2) That the ordinary specific-gravity flask with perforated stopper be replaced by the Sprengel tube or a modification thereof.

(3) That the flask or tube have a capacity of not less than 100 cc when the amount of the sample and the capacity of the balance will permit; in other cases a vessel of 50 cc capacity may be used.

(4) That the standardization of flasks by calculation be limited to those made of glass, of which the coefficient of cubical expansion is known with precision to the sixth decimal place.

Further, I recommend that this association do now make the following change in the method for standardizing flasks for the determination of the specific gravity of fats and oils:

Bulletin No. 46. revised edition, page 51, under "Optional method of standardizing flasks," insert the following in place of the method there given:

The following formula may be used for calculating the weight of water (W^{T°) which a given flask will hold at T° (weighed in air with brass weights at the temperature of the room) from the weight of water (W^t) (weighed in air with brass weights at the temperature of the room) contained therein at t°:

$$W^{T^\circ}=W^{t^\circ}\frac{d^{T^\circ}}{d^{t^\circ}}\left[1+\gamma\,(T-t)\right]$$

d^T=the density of water at T°.
d^t=the density of water at t°.
γ=the coefficient of cubical expansion of glass.

SATURDAY—EVENING SESSION.

The meeting was called to order by the president at 7 o'clock.

The report of the special committee on the subdivision of food work was presented as follows:

REPORT OF THE SPECIAL COMMITTEE ON THE SUBDIVISION OF FOOD WORK.

Resolved, That it be made the duty of the referee on liquor and food adulteration to associate with him such members of the association as he may see fit, each of whom shall be instructed to investigate and report on one or more of the following subjects:

(1) Meat and fish.
(2) Fats and oils (not including dairy products or their substitutes).
(3) Cereal products.
(4) Infants' and invalids' foods.
(5) Saccharine products (including confectionery).
(6) Vegetables (canned, dried, or otherwise preserved).
(7) Tea, coffee, and cocoa.
(8) Spices and condiments.
(9) Vinegar.
(10) Flavoring extracts.
(11) Fruit products.
(12) Fermented and distilled liquors.
(13) Baking powder and baking chemicals.
(14) Preservatives.
(15) Dyes.

Adopted.

The president next called for the report of the committee on recommendations.

REPORT OF COMMITTEE ON RECOMMENDATIONS.

ASH.

(1) The committee recommends the use of calcium acetate and burning ash in some way to prevent volatilization.

Adopted.

CATTLE FEEDS.

(1) Phloroglucin method (Bul. 46, rev. ed., p. 25):

(*a*) The 5th line be changed so as to read: "regulated so as to distill 30 cc in about ten minutes, the distillate passing through a small filter paper."

Adopted.

(*b*) In the 6th line, after "dilute acid," the following words be added: "added in such a manner as to wash down the particles adhering to the sides of the flask."

Adopted.

(*c*) The distillation be continued until the distillate amounts to 360 cc.

Adopted.

(*d*) In the 8th line the words "free from diresorcol" be changed to "purified if necessary."

Adopted.

(*e*) In the 14th line the words "over night" be changed to "at least twenty hours."

Referred to referee for ensuing year.

(*f*) The method of purifying the phloroglucol devised by Mr. Fraps[1] be adopted and added to the paragraph entitled, "Qualitative test of the purity of the phloroglucol." (Bul. 46, p. 26.)

Adopted.

(2) Galactan method (Bul. 46, p. 26): It is recommended that in the fourteenth line, after "ammonia," the following be added: "The filter paper and contents are then washed several times with hot water by decantation, the washings being passed through a filterpaper, to which the material is finally transferred and thoroughly washed.

Adopted.

(3) It is finally recommended that the terms "phloroglucol," "diresorcol," and "furfural" be adopted.

Adopted.

CHANGES IN BULLETIN 46, REVISED.

(1) Page 47, in paragraph 2 (*a*), "soluble acids," insert in the third line the following sentence: "Or the saponification may be conducted in a flask connected with a reflux condenser."

Adopted.

(2) On page 52, sixth line from bottom, add the sentence: "These discs should be prepared a day, or at least a few hours, before using."

Adopted.

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[1] See p. 112.

It was moved and adopted that the question of the number of hours the discs should be made before using be referred to the referee for the ensuing year.

(3) On page 48, eighth line from bottom, change the words "between 1 and 2 grams" to "approximately 2.5 grams." At the end of the same sentence, insert (after a semicolon) the words "or the saponification may be conducted in a flask with a reflux condenser."

Adopted.

It was moved and adopted that the secretary be requested to insert in the next edition of the methods the latest edition of the revision of the atomic weights of the committee on atomic weights of the American Chemical Society.

By vote of the association, the editor was authorized to change the quantities of sodium thiosulphate (p. 50, Bulletin 46) to meet new atomic weights.

DAIRY PRODUCTS.

(1) The committee recommends that paragraph 3, p. 55 of Bulletin 46, entitled "Official optional method for determining casein in milk," be changed to read as follows:

3. *Official optional method for determining casein*—To 10 cc of milk add 50 cc of distilled water at 40°, then add 2 cc of alum solution saturated at 40° or higher. Allow precipitate to settle, transfer to a filter, and wash. The washed precipitate and filter paper are digested as in regular Kjeldahl method, as outlined in the official method, paragraph 1. Calculate casein by multiplying the amount of nitrogen found by 6.25.

Adopted.

(2) The committee recommends that paragraph 4, page 55, of Bulletin 46, entitled "provisional optional method for determining albumin in milk," etc., be changed to read as follows:

Provisional optional method for determining albumin in milk—To the filtrate from the preceding add 10 cc of 20 per cent tannin solution, heat, filter, wash with distilled water, and determine nitrogen by the Kjeldahl method. Multiply the result by 6.25 for albumin

This recommendation went over without action.

The recommendations made by Mr. Ewell[1] relative to the determination of the specific gravity of fats and oils, as well as the change recommended under the "Optional method of standardizing flasks," Bulletin 46, revised edition, p. 51, were favorably reported and adopted.

Mr. Carr then presented the report on tannin.

REPORT ON TANNIN.

By Oma Carr, *Referee.*

Owing to disappointment in not receiving a hide-powder suitable for the official method, the referee delayed sending out the samples for cooperative work until November 1. On that date samples of liquid oakwood extract and solid quebracho extract, selected as representing typical commercial materials, were sent to the collaborators. Reports were received from eight analysts, giving the results to be discussed in the following pages. Work done under the referee's direction will be entered first, to be followed by a tabulation of the collaborative analyses.

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[1] See p. 125.

SOLUBLE SOLIDS.

In nearly all tanning materials the anhydrids, gums, phlobaphenes, etc., grouped as insolubles, are present in the analysis solution in such state of fine division that filtration, even through finest paper, will not effect complete removal. In such cases the method permits the use of kaolin, previously washed in a portion of the solution, with the view of "filling" the paper and thus obstructing the passage of the insoluble matter. While this procedure accomplishes the object of a clear filtrate, it is open to the objection that kaolin is of varying composition and that the increased time of contact with the paper increases the absorption and lowers the result as to soluble solids.

With the aim of overcoming the paper absorption, the referee tried various methods of mordanting the paper and coloring with a dye without much success beyond pointing the way for further experiment. The paper was saturated with a solution of gallotannic acid and struck with a basic dye. Looseness of attraction prevented a good result, and subsequent washing removed a large amount of color. Paper so prepared, however, possessed less absorption power than the untreated fiber and the filtering power remained good. Possibly further experiment may enable the paper makers to produce an absorption-free fiber. As the fiber of the paper acts basically in tannin attraction, it was thought that the addition of a volatile acid, inert as to chemical action on the constituents of the solution, might overcome the attraction. Experiments were conducted on a solution of oakwood extract, adding 0.36 gm of acetic acid, in weak solution, to each 100 cc before filtration, and passing through plain and pleated No. 590, 15 cm paper.

Effect of the addition of acetic acid.

Volume of filtered solution taken for determination.	Soluble solids.	
	Plain paper.	Pleated paper.
	Per cent.	*Per cent.*
First 50 cc	41.01	41.90
Second 50 cc	41.17	41.95
Third 50 cc	41.66	42.27

It will be noted that the rate of filtration was higher with the pleated paper.

The action of plain folded paper, without acid, is shown in the following table, and it will be seen that there was a decrease rather than an increase in the rate of filtration:

Soluble solids obtained without the addition of acetic acid and the use of plain folded paper.

Method of filtration and volume of filtered solution taken.	Soluble solids.
	Per cent.
75 cc through, 50 cc taken	41.07
75 cc through same paper, 50 cc	40.95
75 cc through same paper, 50 cc	40.79

The action of kaolin and barytes, with the same solution, and no acid gave the following results:

Comparison of effect of kaolin and barytes on the amount of soluble solids obtained.

Method of filtration.	Soluble solids.
	Per cent.
150 cc passed, then 5 gm kaolin, washed in solution	39.52
150 cc passed, then 10 gm kaolin, washed in solution	37.34
150 cc passed, then 5 gm barytes, washed in solution	40.79
150 cc passed, then 10 gm barytes, washed in solution	40.49

The use of a 100 cc solution, 0.02 gm of lead acetate, 0.36 gm of acetic acid, correcting for removal by lead and dilution, and filtering through No. 590, 15 cm, pleated paper, gave triplicate determinations, as follows: 41.92, 41.70, 41.85 per cent soluble solids. The filtrates were brilliantly clear.

Work was undertaken along the same lines with solid quebracho extract, with the following results:

Comparison of effect of kaolin and barytes on the amount of soluble solids obtained, using plain folded paper.

Method of filtration.	Soluble solids.
	Per cent.
150 cc passed, then 5 gm kaolin, washed in solution	79.82
150 cc passed, then 10 gm kaolin, washed in solution	74.67
150 cc passed, then 5 gm barytes, washed in solution	76.36
150 cc passed, then 10 gm barytes, washed in solution	76.80

Effect of the addition of acetic acid, using pleated paper.

Volume of filtered solution taken for determination.	Soluble solids.
	Per cent.
First 50 cc	77.05
Second 50 cc	76.82
Third 50 cc	76.71

The filtrates were clear.

By the use of 100 cc of solution, 0.02 grams lead acetate, filtered through asbestos, and corrected for lead removal and dilution, triplicate determinations were obtained as follows: 74.41, 74.38, 74.40 per cent soluble solids. The filtrates were brilliantly clear.

The use of 100 cc solution, 0.02 gm acetate lead, 0.32 gm acetic acid, filtered through No. 590, 15 cm, pleated, and corrected for removal by lead and dilution gave the following triplicate determinations: 74.95, 75.24, 75.15 soluble solids. The filtrates were brilliantly clear.

A review of these results develops the following conclusions:

(1) Filtration through paper, plain or pleated, without "assistants" does not totally remove insolubles.

(2) Absorption is largely a function of the time the solution is in contact with the paper, being less for free filtering and greater for viscous solutions.

(3) Acetic acid modifies the basic nature of the paper.

(4) It is possible to secure concordant results by the use of lead acetate, acetic acid, and paper.

(5) As the method stands for soluble solids, it gives low figures for the oakwood and high for the quebracho, and this will hold good in the comparison of any materials so differing.

The referee is of the opinion that results reported to this association in the past, and the experience of the International Association of Leather Trades Chemists, are ample support for the retention of Schleicher and Schüll, No. 590, 15 cm, for all filtrations. It will be found, however, in substituting the pleated for the simple folded form now stipulated, that the passage of the liquor is expedited and the absorption is reduced.

Finally, with reference to the subject of soluble solids, the referee would suggest the following optional method and the substitution of "barytes" for "kaolin" in the official method:

> To 100 cc of the solution add 10 cc of a solution of acetate of lead containing 4 grams of the salt per liter, adding the reagent drop by drop from a burette and stirring meanwhile. Now add 10 cc of a solution of acetic acid containing 36 grams glacial acid per liter, stirring. Throw on double-pleated filter, reject until clear, evaporate 50 cc, and dry.
>
> To another portion of 100 cc repeat the foregoing, except that 20 cc of lead acetate solution shall be used.
>
> Residue from first treatment shall be multiplied by 1.2, and that from the second by 1.3, to bring back to the original 100 cc of solution. Add to the corrected first residue the difference between corrected first and second residues, and calculate as soluble solids. This corrects for the dilution and removal by the lead salt.

HIDE POWDER.

Events of the past summer have emphasized the necessity that hide powder possess the qualifications specified by the method. The principal importer of Vienna powder has supplied the referee with samples from four different lots of powder, none of which approach the standard of insolubility and absorption power. The Vienna powder, generally used because of its close adherence to the limits of insolubility and absorption, has recently shown such wide departure as to fall wholly without the range of allowable variation.

Inasmuch as the determination of tannin is dependent upon the character of the powder employed, no analysis can be truthfully reported as by the official method unless the powder employed shall fall within the prescribed limits. This distinction between powders within and without these limits is not only essential but vital to the value of results, from either the scientific or commercial standpoint. Many of the largest consumers of tanning materials base heavy contracts as to quality upon the official method, and unless it shall be closely stipulated that the powder employed shall answer the requirements of the method, something worse than controversy may result.

The referee, therefore, recommends that to Section VIII, page 80, shall be added the following paragraph:

"(*c*) Any analysis made with a powder which does not fulfil the conditions of the preceding paragraphs, shall not be reported as by this method."

The referee has made several experiments with powders falling without the requirements of the method, with results emphasizing the necessity for the foregoing recommendation.

ACID HIDE POWDERS.

The four powders considered in the following table were markedly acid, and when suspended in water preparatory to an analysis softened into a jelly-like mass which resisted the drying action of squeezing in linen. In order to remove the acidity of the powder it was necessary to use considerable quantities of sodium carbonate in the washing water:

Determination of nontannins using acid hide powders.

Methods.	Nontannins.
	Per cent.
Powder I:	
Without carbonate	16.93
With carbonate, washed three times	16.34
With carbonate, official	16.23
Powder II:	
Official	17.89
With carbonate, official	17.05
Powder III:	
Without carbonate, official	18.31
With carbonate, official	17.12
Powder IV:	
Without carbonate, official	17.68
With carbonate, official	16.74

When it is necessary to resort to an expedient, such as delicate neutralization, it is likely that different operators would return widely ranging results.

WASHING THE POWDER.

A good powder was used and washed with the following results:

Effect of variations in washing on the amount of nontannins obtained.

Number of washings.	Nontannins.
	Per cent.
Not washed	19.74
Washed once in shaker	17.90
Washed twice in shaker	18.04
Washed three times in shaker	17.96
Washed four times in shaker	17.98

VARYING QUANTITIES OF HIDE POWDER.

A large quantity of powder was washed and pressed and duplicate determinations made with increasing quantities of wet pressed hide, which contained 75 per cent of moisture:

Determination of nontannins with increasing quantities of hide powder.

Amount of hide powder.	Nontannins.	Mean.
	Per cent.	
50 grams wet hide	17.33 17.34	17.34
60 grams wet hide	16.61 16.63	16.62
70 grams wet hide	16.11 15.96	16.04

Inasmuch as the moisture content of the wet pressed hide is variable with the physical condition thereof, a definite statement of the dry hide present in the wet pressed cake must be made. Results are concordant for any definite quantity, but the fineness of some powders permits great loss in squeezing, and the actual dry hide present in the solution thereby becomes variable. The referee thinks it wise to stipulate that the dry hide present in the wet cake taken for the determination shall fall between 12 and 13 grams.

VOLUME OF SOLUTIONS FOR DRYING.

Owing to the sensitiveness of tanning materials to the heat employed in drying, it is believed that the use of volumes yielding residues of from 400 to 500 milligrams is preferable to volumes giving greater residues. It is believed that if, instead of 100 cc 50 cc shall be used for total and soluble solids the resultant residue may be dried to constant weight in less time and at less risk of decomposition or oxidation. The experience of the referee is that residues of 400 milligrams may be dried to nearly constant weight on the steam or water bath in five hours, passing through an air or water oven for a half hour to remove surface moisture. While some laboratories are equipped to use volumes of 100 cc, others are not and it is believed that the smaller volume and residue will be advantageous in bringing the entire determination within one working day.

The referee would recommend, therefore, the substitution of 50 cc wherever in the method 100 cc is stipulated for drying.

Summary of analyses.

OAKWOOD EXTRACT.

Analyst.	Total solids.	Soluble solids.	Insoluble.	Tannin.	Nontannin.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	43.45	42.24	1.21	25.64	16.60
2	43.37	42.54	.83	25.78	16.76
3	42.74	42.13	.61	25.20	16.93
4	42.53	40.65	1.88	24.72	15.93
5	42.85	42.22	.63	24.07	18.15
6	43.23	42.53	.70	25.07	17.46
7	42.94	42.29	.65	25.25	17.01
8	43.29	42.26	1.03	25.11	17.15
Averages	43.05	42.11	.94	25.11	16.99
Maxima	43.45	42.54	1.88	25.78	18.15
Minima	42.53	40.65	.61	24.07	15.93

QUEBRACHO EXTRACT.

1	87.33	74.00	13.33	64.70	9.30
2	86.71	74.52	12.19	65.56	8.96
3	87.08	76.33	10.75	66.84	9.49
4	90.13	79.82	10.31		
5	87.45	75.80	11.65	67.08	8.72
6	87.40	76.29	11.11	68.35	7.94
7	85.98	75.10	10.88	67.47	7.63
8	86.45	74.45	12.00	66.25	8.20
Averages	87.32	75.70	11.59	66.61	8.61
Maxima	90.13	79.82	13.33	67.47	9.49
Minima	85.98	74.00	10.31	64.70	7.63

The above eight analyses exhibit some improvement over the results previously obtained by collaboration. The quebracho is a severe trial on the accuracy of the method, owing to the high tannin and the extreme fineness of the insolubles in the analysis solution. Variations in nontannins may be largely attributed to variations in character of hide powder used, as the analysts used the powder employed in their respective laboratories. Given a soluble solids determination accurate to 1 per cent on a solid extract, it is likely that the principles of the nontannin determination as now laid down in the method may be considered satisfactory. The range from maximum to minimum is relatively greater for all determinations, except nontannins, in the oakwood than in the quebracho.

THE METHOD.

The following modifications of the official method, Bulletin 46, revised, page 79, VIII, Methods for the Analysis of Tanning Materials, are recommended:

III. Moisture. (*b*) 2. Read "For eight hours at 100° to 110° in an air bath."

IV. Total solids. For "100 cc" read "50 cc."

V. Soluble solids. For "double-folded filter" read "double-pleated filter." For "100 cc" read "50 cc." For "kaolin" read "barytes."

Add paragraph giving optional method, as follows:

Optional method.

(*a*) To 100 cc of the solution add 10 cc of a solution of lead acetate (4 grams per liter), adding the reagent drop by drop from a burette and stirring meanwhile. Add 10 cc of a solution of acetic acid (36 grams glacial acid per liter), stirring. Throw on double-pleated filter, reject until clear, and evaporate and dry 50 cc.

(*b*) On another portion of 100 cc repeat the foregoing, except that 20 cc of lead acetate solution shall be used.

Residue from (*a*) shall be multiplied by 1.2, and from (*b*) by 1.3, to bring back to the original 100 cc. Add to the corrected weight of the residue from (*a*) the difference between (*a*) and (*b*) and calculate the found residue to soluble solids. This corrects for dilution and removal by lead.

VI. Nontannins. For lines 6–9 substitute the following: "Weigh the remaining three-quarters, which must contain between 12 and 13 grams of dry hide; add to 200 cc of the solution and shake ten minutes. Throw on funnel with cotton plug in stem, return until clear, evaporate 50 cc, and dry."

VIII. Testing hide powder. (*a*) For "100 cc" read "50 cc," and for "10 mg" read "5 mg." (*b*) For "100 cc" read "50 cc."

Add paragraph (*c*), as follows:

(*c*) Any analysis made with a powder which does not fulfill the conditions of the preceding paragraphs shall not be reported as by this method.

At this stage of the proceedings, Mr. Krug presented the following papers:

REPORT ON COMPARATIVE HIDE-POWDER TESTS.

By William H. Krug.

During the last year various chemists engaged in the analysis of tanning materials have frequently complained of the character of the hide powder they were using, and have ascribed unsatisfactory results to variations in its absorbent power. The hide powder generally used in this country is made by the Vienna Experiment Station, and has, until recently, given almost universal satisfaction. Within the last year, however, it has undergone marked changes in that it is quite acid in reaction and retains a large amount of moisture after being squeezed even when repeatedly washed with water. When neutralized with sodium carbonate it loses this power of retaining water, but also decreases in its power of absorption with reference to tannin. It is generally claimed that this acid hide powder when used without previous neutralization gives a figure for nontannins which is too low, and thus in turn results in too high a tanning value being ascribed to the material under examination.

It being evident that this matter is of great importance to all chemists interested in this branch of analytical chemistry, I some time ago sent a sample of quebracho extract, which had been ground and carefully mixed so as to render it strictly uniform in composition, to Mr. Alsop, of the United States Leather Company, and Mr. Teas, of the Elk Tanning Company. The sample was accompanied by a sufficient quantity of Vienna hide powder to make several determinations, and I suggested that the extract be analyzed both with and without previous neutralization of the powder with sodium carbonate. Unfortunately Mr. Teas was unable, through pressure of other work, to make more than one analysis in which he used the hide powder on hand at his laboratory. As this was derived from the same source as that sent by me for the comparative work, the two powders are probably identical. Mr. Alsop sent quite a complete report embodying results obtained both with hide powder in use at his laboratory and that sent him. The results are contained in the following table:

Comparative analyses of quebracho extract both with and without previous neutralization of hide powder.

No.	Analyst.	Method employed.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Remarks.
			Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	
1	Alsop...	Laboratory hide, official method, not neutralized.	85.68	76.74	8.94	10.25	66.49	Showed traces of tannin in nontannin filtrate. Filtrate was clear.
2	....do...	Laboratory hide, official method, neutralized.	85.68	76.74	8.94	12.74	64.00	Filtrate was turbid.
3	....do...	Laboratory hide, official method, treated with one-third the amount of Na_2Co_3 necessary to neutralize.	85.68	76.74	8.94	9.78	66.96	Filtrate was clear and contained no tannin.
4	....do...	Hide powder sent for comparative test, not neutralized.	85.68	76.74	8.94	10.68	66.06	Hide powder washed according to official method.
5	....do...	Hide powder sent for comparative test, treated with one-third the amount of $Na_2 Co_3$ necessary to neutralize.	85.68	76.74	8.94	10.79	65.95	Hide powder washed according to official method.
6	....do...	Laboratory hide, not neutralized.	85.68	76.74	8.94	10.45	66.29	In these analyses the hide powder was washed according to the method adopted in Mr. Alsop's laboratory.
7	....do...	Laboratory hide, treated with one-third the amount of $Na_2 Co_3$ necessary to neutralize.	85.68	76.74	8.94	9.90	66.84	
8	....do...	Laboratory hide, neutralized.	85.68	76.74	8.94	12.14	64.60	
9	Teas....	Laboratory hide.	84.95	75.05	9.90	10.65	64.40	Treatment of hide powder not stated.
10	Krug...	Hide powder sent for comparative test, not neutralized.	85.35	77.43	7.92	10.08	67.35	20 grams hide powder used, washed according to official method.
11	....do...	Hide powder sent for comparative test, neutralized.	85.35	77.43	7.92	11.74	65.69	20 grams hide powder used, washed in a beaker ten times by decantation, using a liter of water each time. Nontannin filtrate showed traces of tannin and was turbid.

Comparative analyses of quebracho extract both with and without previous neutralization of hide powder—Continued.

No.	Analyst.	Method employed.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Remarks.
			Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	
12	Krug ...	Hide powder sent for comparative test, neutralized.	85.35	77.43	7.92	10.49	66.94	30 grams of hide powder used, washed in a beaker ten times by decantation, using a liter of water each time. Nontannin filtrate showed traces of tannin and was turbid.
13	....do ...	Hide powder sent for comparative test, neutralized.	85.35	77.43	7.92	9.81	67.62	40 grams of hide powder used, washed in a beaker ten times by decantation. using a liter of water each time. Nontannin filtrate was clear and showed no tannin.

Mr. Alsop describes the method of washing the hide powder in use in his laboratory as follows:

> Enough hide to make the determination is treated with about one hundred times its weight of water. If too acid to squeeze well, one-third the amount of sodium carbonate necessary to neutralize is added. It is then squeezed by hand through linen, treated with the same amount of water, and squeezed once more. If sodium carbonate has been added this washing is repeated again. It is then squeezed in a press and 50 grams taken for nontannins. A portion is dried for determination of moisture. I can obtain more uniform results by this method than by the official method where 20 grams are taken for each determinatinon.

A comparison of the results obtained by Mr. Alsop and myself shows a close concordance with reference to the nontannins when the hide powder was not neutralized, and this is true both of the hide powder sent him for the comparative work and that in use in his own laboratory. When the acidity of the hide powder is neutralized, however, its absorption power becomes less, the nontannin filtrates are turbid, and show traces of tannin. It was found that after neutralization it requires at least 40 grams of hide powder to obtain a filtrate free from tannin. The use of such a quantity is out of the question on account of the cost of the material.

The results clearly show the influence which the acidity of the hide powder exercises on the percentage of nontannins obtained and render it evident that something should be done to enable chemists to obtain a neutral powder. While Mr. Alsop's method of treating with one-third the amount of sodium carbonate necessary for neutralization may be good, the question nevertheless arises if this is not merely choosing a mean between two evils. The accuracy of the official method depends to a very great extent on the character of the hide powder used, and every precaution should be employed to avoid sources of error in this feature of the method. I would therefore suggest that the matter be taken up by the association, possibly by inserting a clause in the present method, directing that the hide powder used in the determination of the nontannins shall be neutral. The method as it now stands specifies certain standards as to the maximum amount of soluble matter which shall be present and the absorption power. It appears, however, that these restrictions do not cover fully the exigencies of the case.

The following paper was then presented:

A COMPARISON OF THE INTERNATIONAL FILTER-TUBE METHOD AND THE OFFICIAL HIDE-POWDER METHOD.

By H. W. Wiley and William H. Krug.

The filter-tube method has been employed for many years by chemists on the continent in the analysis of tanning materials. It was officially adopted by the International Association of Leather Trades Chemists at a conference held in England in 1897, and has since been retained by that association. The objection made by American chemists to this method is that it yields a figure for nontannins which is too low, and thus gives the tanning material a higher value than it should have. At the last meeting of the international association, which was held in Paris last summer, and which one of us attended as American delegate, it was arranged with Professor Procter to make a series of comparative tests on tanning materials which were to be furnished by him. These materials comprised a sample each of oak-wood extract, chestnut-wood extract, liquid quebracho extract, mimosa extract, solid quebracho extract, sumac, valonia, golden wattle, and mimosa bark. Upon arrival of the samples it was found that the bottle containing the liquid quebracho extract was broken, and the analyses of the sumac and golden wattle were interrupted by the breaking of the bottles during extraction. The analyses by the filter-tube method were made in Professor Procter's laboratory, while those by the official method were made in the Division of Chemistry, United States Department of Agriculture. The results obtained are compiled in the following tables:

Comparison of results obtained by the official and the filter-tube methods in the analysis of tanning materials.

Constituents.	Oak-wood extract.		Chestnut-wood extract.		Mimosa extract.		Solid quebracho extract.	
	Filter-tube method.	Official method.	Filter-tube method.	Official method.	Filter-tube method.	Official method.	Filter-tube method.	Official method.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Moisture	59.60	59.50	59.80	60.36	50.80	50.28	....	16.84
Total solids by dilution	....	40.34	....	39.74	....	49.90	81.10	83.39
Soluble solids by dilution	....	40.02	....	38.89	....	49.70	69.10	75.20
Reds	0.40	0.32	0.80	0.85	0.00	0.20	12.00	8.19
Nontannins	13.00	14.66	10.60	13.11	9.00	12.33	....	14.01
Available tannins	27.00	25.36	28.80	25.78	40.20	37.37	....	61.19

Constituents.	Valonia.		Mimosa bark.	
	Filter-tube method.	Official method.	Filter-tube method.	Official method.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Moisture	12.20	12.60	10.80	....
Total solids in extract[1]	....	53.51	....	47.04
Soluble solids in extract[1]	....	52.69	....	43.30
Reds in extract[1]	....	0.82	....	3.74
Nontannins	12.90	13.20	10.70	10.11
Available tannins	40.30	39.49	32.30	33.19
Total insoluble matter	34.60	34.71	46.20	....

[1] Calculated to original material.

The results show in general a very good agreement as to the amount of moisture present in the samples. The same is true of the reds, except with the solid quebracho extract, where the high percentage obtained with the filter-tube method shows the effect of rapid cooling of the diluted extract, as specified by this method, upon the sensitive tannin which it contains. The chief point of interest in these figures is naturally the nontannins, as these indicate the variation caused by the different application of the hide powder. In every case except one, that being mimosa bark, the filter-tube method gives lower nontannins than the official method and correspondingly higher tannins. It is evident, therefore, that a true international method is an absolute necessity. Large quantities of tanning materials are annually sold to American importers, the European merchant demanding payment on the basis of the results obtained by the filter-tube method. The American importer in turn is forced to sell to the tanner on the basis of figures obtained by the official method, the result being constant friction between buyer and seller. It gives us pleasure to state that the first step toward a uniform method has been taken, the International Association at its last conference in Paris having adopted the method of cooling the diluted extracts used in this country. It is to be hoped that the efforts in this direction will be continued, and will result finally in the development and universal adoption of a truly international method for the analysis of tanning materials.

Mr. Sawyer gave a very interesting talk on various debatable points in the present method for the analysis of tanning materials. He advocated the use of a smaller volume of the solutions in determining total and soluble solids, and suggested a series of comparative experiments on various methods of filtration.

Mr. Wiley. I attended the International Congress of Leather Chemists, which met in Paris in August, 1900. The president of this congress, Prof. H. R. Proctor, is well known to chemists in this country, especially those engaged in the tanning industries.

I was most cordially received as the representative of the Association of Official Agricultural Chemists and invited to a seat of honor at the president's right, and asked to take part in the discussion. Finding that there was no American representative in the congress, I decided to become a member thereof, in order that we might be not only officially but actively represented. The proceedings of the congress were conducted in three languages, which made the sessions rather monotonous. Each member of the congress spoke in his native language, and his speech was afterwards repeated in the other two languages of the congress, French, German, and English being the three languages recognized.

In adopting methods of research and analysis the processes recommended by the Association of Official Agricultural Chemists received due consideration. In fact, an earnest effort was made to harmonize the methods employed on the Continent with those employed by our own chemists. Some of our methods were adopted en bloc and others in a more or less modified form. It is evident that with a very little effort we may be able to unify the methods in vogue both in America and in Europe. This would be extremely useful, not only from a scientific point of view, but especially from the commercial aspect, since in this case analyses made by chemists on the two continents would be conducted by the same methods and be strictly comparable.

The banquet tendered the congress by the syndicate of leather merchants of Paris was an elaborate affair given at the Hotel Continental, and was one of the very pleasing features of the meeting.

Mr. Wiley then spoke on the question of the unification of methods of fertilizer analysis as follows:

The international committee on the unification of methods of fertilizer analysis presented its report to the Fourth International Congress of Applied Chemistry, which met in Paris in July, 1900. The president of this commission was Dr. Ritter von Gruber, and I had the honor to be the American member.

A basis of international agreement for fertilizer analysis was adopted by the committee, in which all the leading principles were recognized as standard and the details of manipulation were left open for further consideration. The committee considered that it was a wiser plan at this period to agree upon the essential principles, and to cite the standard authorities of the United States, Germany, and France for the details at the present time. The report was printed in three languages, German, French, and English, stating the essential points of agreement and citing the standard authorities of different countries. This report of the committee was presented to the section of agricultural chemistry, and, after a prolonged discussion, it was unanimously adopted.

This is a matter for congratulation on the part of the Association of Official Agricultural Chemists, because it was a recognition by this international congress of the valuable work which has been accomplished by this body, and the official methods of analysis are now for the first time cited in an authoritative publication in Europe for the guidance of analysts throughout the world.

The report of the committee on recommendations was now called for.

REPORT OF COMMITTEE ON RECOMMENDATIONS.

TANNIN.

The committee recommends the adoption of the amended method for tannin, as given by Mr. Carr in his report. (See p. 133.)

Adopted.

Mr. Sawyer suggested that the referee for the ensuing year investigate the evaporation of smaller volumes of solution than 50 cc and weighing of smaller residues than 0.4000 gram.

Adopted.

Mr. Sawyer also moved that the referee for the ensuing year study the process of sedimentation and of centrifuging, to replace filtration.

Adopted.

Mr. Huston suggested that, in connection with the above study, the referee try filtering through a Pasteur filter instead of paper.

Adopted.

The referee on insecticides being absent, the report was read by the secretary, as follows:

REPORT ON FUNGICIDES AND INSECTICIDES.

By Louis A. Voorhees, *Referee.*

In presenting the following report your referee begs to say a word of explanation and apology. Having been continued as associate, he awaited the pleasure of the referee, but heard nothing until suddenly informed that the latter, for excellent reasons, would be unable to conduct the work, which therefore would devolve upon the writer. This occurred in the spring, when the large amount of work in the fertilizer control was beginning to require the entire attention for the summer of the chemical force at the New Jersey experiment station. This state of affairs was submitted to the president of the association, and it was accordingly agreed that since it would be impossible to present any analytical work, the report should consist of a further compilation of methods.

A year ago the writer, in his report as associate referee, submitted methods for the examination of fungicides and insecticides, which were adopted as provisional methods by the association. The methods were selected entirely by the writer. This year it was deemed advisable to get the opinion of the station workers, and a circular letter was sent to all the experiment stations and to several commercial laboratories, as follows:

> Dear Sir: The undersigned, owing to the resignation of Dr. de Schweinitz as referee of the Association of Official Agricultural Chemists on fungicides and insecticides, has been appointed by the president to undertake the work.
>
> Last year, as associate referee, and in the absence of Dr. de Schweinitz in Europe, the undersigned proposed certain methods for the determination of the essential constituents of the various fungicides and insecticides, which methods were adopted provisionally by the association, and published in the annual proceedings for 1899, page 101.
>
> Will you kindly advise the writer if you have employed any of these methods in your laboratory at any time, and with what success? Also please point out any difficulties met with or modifications proposed, or give a detailed statement of your own methods for these determinations. The results of any analytical work, comparative or otherwise, will be appreciated, and due credit given in every instance.
>
> The determinations involved are:
>
> - Alkalinity in potash and soda lyes.
> - Arsenic in arsenicals.
> - Copper in copper carbonate.
> - Cyanogen in potassium cyanid.
> - Formaldehyde in formalin.
> - Nicotin in tobacco.
>
> Hoping to receive an early reply, I remain,
>
> Yours very truly,
> Louis A. Voorhees,
> *Referee Association of Official Agricultural Chemists*
> *on Fungicides and Insecticides.*

Twenty-seven replies were received, 14 stating that no work has been done along these lines, and the remaining 13 communicating more or less extensive results of work and experience. The latter were received from the following-named persons:

E. E. Ewell	U. S. Department of Agriculture, Washington, D. C.
E. W. Hilgard	California Experiment Station.
A. L. Winton	Connecticut Experiment Station.
S. Avery	Idaho Experiment Station.
A. M. Peter	Kentucky Experiment Station.
H. J. Patterson	Maryland Experiment Station.
C. A. Goessman	Massachusetts Experiment Station.
N. E. Wilson	Nevada Experiment Station.
M. B. Hardin	South Carolina Experiment Station.
C. H. Jones	Vermont Experiment Station.
R. J. Davidson	Virginia Experiment Station.
Elton Fulmer	Washington Experiment Station.
Wm. S. Myers	Rutgers College.

No methods were communicated for the determination of alkalinity in lyes or soaps or of cyanogen in potassium cyanid. Mr. R. J. Davidson used Kissling's method for nicotin, while Mr. A. M. Peter, on the other hand, writes as follows:

> Recently I have made a determination of nicotin in tobacco and tobacco extracts, in which I used the Kissling method and also the modification proposed by Winton and the Lloyd method. It seems that the Lloyd method gave as good results as either of the others and was much more rapid and easily worked and much better adapted to technical work. In this connection I have been trying luteol as indicator in titrating the nicotin, and I believe it is better for this purpose than litmus, being apparently much sharper in its indications.

A. L. Winton calls attention to the method for the determination of formaldehyde, which is described on page 143 of the Connecticut Report for 1899.

With these exceptions, the replies received related to the determination of arsenic in arsenicals, especially paris green, the separation of the arsenic and the allied determinations of copper, acetic acid, water-soluble arsenic, and lead.

For the separation of the arsenic from copper, Goessman and Avery precipitate the copper as suboxid; Goessman (alternate method) and Myers precipitate the mixed sulphids and separate with sodium sulphid, and Peter and Patterson determine the arsenic by precipitation as magnesium pyroarsenate without separation, the copper passing into the filtrate. C. H. Jones also precipitates directly or with previous separation with ammonium molybdate. He also communicates the separation of arsenic from lead, which is particularly applicable in the analysis of arsenate of lead.

The determination itself of the arsenic is by oxidation and precipitation as magnesium pyroarsenate in the laboratories of Peter, Goessman, Fulmer, Patterson, Hardin, and Jones; S. Avery uses the volumetric method of Thorn Smith, and Myers and Wilson use the method proposed by the writer on page 105 of the proceedings of this association for 1899.

The determination of the copper is reported by Goessman to be as copper oxid, by Myers as copper sulphid (the method proposed in 1899), and by Fulmer and Avery by titration with potassium cyanid.

Professor Goessman sends the method for the determination of the acetic acid in paris green, and the preliminary tests are from Professor Hilgard's bulletin, No.126 of the California station.

In regard to the examination of paris green, the most extensive and systematic work that has come to the writer's notice is that of J. K. Haywood, of the U. S. Department of Agriculture, which appeared in the journal of the American Chemical Society for September, 1900, and to which the attention of the referee was called by E. E. Ewell, at that time acting chemist of the Department. It is impracticable to quote from Mr. Haywood's discussion of the several methods which he tried, for it is all of such value and importance that in order to quote at all, it would be necessary to reproduce the article in full. The methods on which he finally determines are given, and those who wish to use them are urged to consult the original article.

METHODS.

ALKALIES AND ALKALINITY.

Any of the standard methods may be used, both for the alkalies and for the alkalinity of the ash of the various soap preparations.

ARSENIC IN ARSENICALS.

Preliminary examination.

See Bulletin 126, California Agricultural Experiment Station, p. 12.

Chemical examination.

(1) *Hydrogen sulphid method.* See Bulletin 57, Division of Chemistry, United States Department of Agriculture, pp. 104, 105.

(2) *Determination as pyroarsenate.*[1] Weigh out 1 gram of material in a beaker and add 50 cc of water; digest for one-half hour in a boiling water bath with about 5 grams of caustic potash. This reduces the copper to the red suboxid. Filter, wash with water, dry the precipitate, and burn in a porcelain crucible; add a few drops of nitric acid to complete the oxidation, carefully drive off the excess of nitric acid, cool, and weigh.

Oxidize the filtrate containing the arsenic with chlorin gas, let stand for a few hours, and if chlorin gas can be detected by the smell very carefully neutralize the solution with ammonium hydrate and precipitate the arsenic with magnesia mixture; allow to stand at least two hours, filter through Gooch crucible, dry, burn, cool, and weigh, the resulting product being magnesium pyro-arsenate ($Mg_2As_2O_7$). Make the solution containing the arsenic acid with hydrochloric acid and change the arsenious acid to the arsenic acid by the addition of small quantities of chlorate of potash at frequent intervals. Drive off the excess of chlorin by repeated evaporations, make alkaline with ammonium hydrate, and precipitate with magnesia mixture.

(3) *Method of Thorn Smith, modified by J. K. Haywood.*

See Journal of the American Chemical Society, Vol. XXII, No. 9, pp. 576–578.

(4) *Estimation of copper by potassic cyanid* (Parkes and C. Mohr). See Sutton's Volumetric Analysis, pp. 162–163.

(5) *Estimation of acetic acid.* "The figure for acetic acid in paris green is usually obtained by subtracting the sum of total arsenious oxid, water, cupric oxid, and residue left after treating with dilute hydrochloric acid, from 100." [2]

The following method is communicated by C. A. Goessman:

Weigh out 0.6 gram of the paris green, transfer to a flask, capacity about 400 cc Make up a 10 per cent solution of sulphuric acid, and add about 200 cc of this solution to the substance in the flask, affix to the condenser, apply heat and distill over the acetic acid. Collect 125 cc of the distillate and titrate against N/10 soda solution, using phenol-pthalein as an indicator.

The number of cc of N/10 soda solution required to neutralize the acid will give the percentage of acetic acid.

(6) *Estimation of water-soluble arsenic.* Mr. J. K. Haywood communicates the following method:

Weigh 1 gram of paris green, transfer to a 500 cc flask, add 400 cc water at room temperature, and shake with a rotary motion. Allow the flask to stand at room temperature for from twelve to fourteen days, shaking occasionally each day. Finally complete the volume to 500 cc, filter through a dry filter, take 100 cc of the filtrate, and determine the arsenious oxid.

(7) *Estimation of arsenate of lead.* Mr. C. H. Jones, chemist of the Vermont Agricultural Experiment Station, writes as follows:

Below find the outline of a method given me by Mr. F. J. Smith, late chemist of the Massachusetts Gypsy Moth Commission, which is particularly applicable in the analysis of arsenate of lead. I have used it in the estimation of arsenic in the above-named material, and it is apparently satisfactory.

Take 0.5 gram of material and mix in porcelain dish with two parts sodium carbonate and one part powdered sulphur. Fuse at a low temperature for about thirty minutes, and then increase heat if need be until sulphur fumes are driven off. Cool, digest in warm water until dissolved. The lead and arsenic are as sulphids in alkaline solution—arsenic soluble, lead insoluble. Filter, using Gooch crucible, and wash with ammonium acetate solution (3 grams to 200 of water) to prevent lead from running through. The lead in the precipitate may be estimated as desired. Filtrate is evaporated to dryness, oxidized with fuming nitric acid and a trifle of potassium chlorate and hydrochloric acid, and precipitated in ammoniacal solution with magnesia mixture directly (preferred) or with molybdic solution as with phosphoric acid.

It will be noticed that I have given in this compilation the methods in their entirety, as communicated to me or referred to the publications where they are printed. This is especially noticeable in the case of the methods for the analysis of the arsenicals. Perhaps a more systematic, and hence a more scientific, plan would have been to compile all the separations as such, and follow with the methods for the actual determinations, but I have not done so, as this would destroy the individuality of the contributions, and in many cases the particular method of determination employed follows the method of separation as a natural sequence. I submit this compilation to the Association of Official Agricultural Chemists as representing the opinion of its workers, and would respectfully recommend that they all be adopted provisionally for trial and comparison in actual analytical work in the same manner as has in the past so perfected our methods of fertilizer analysis.

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[1] As communicated to the referee by C. A. Goessman.

[2] Journal of the American Chemical Society, Vol. XXII., No. 9, p. 578.

POTASSIUM CYANID.

Determine purity by Liebig's volumetric method. It is based on the fact that when a solution of silver nitrate is added to an alkaline solution containing cyanogen, with constant stirring, no permanent precipitate of silver cyanid occurs until all the cyanogen has combined with the alkali and the silver to form a soluble double salt (in the presence of potash, for example, KCy, AgCy). If the slightest excess of silver over and above the quantity required to form this combination be added, a permanent precipitate of silver cyanid occurs, the double compound being destroyed.[1] If, therefore, the silver solution be of known strength, the quantity of cyanogen present is easily found, 1 eq. of silver in this case being equal to 2 eq. of cyanogen.

FORMALIN.

Qualitative: Use the tests for formaldehyde in milk described on page 113 of the proceedings of the convention of this association in 1897 (Bulletin No. 51, Division of Chemistry).

Quantitative: Method of Blank and Finkenbeiner,[2] of which the following is an abstract:

(1) Three grams of the solution are weighed off into a tall Erlenmeyer flask containing 25 to 30 cc of double normal sodium hydroxid; 50 cc of pure 2.5 to 3 per cent hydrogen peroxid solution are then gradually added during a space of 3 minutes, through a funnel placed in the neck of the flask to prevent spurting. After standing two or three minutes the funnel is washed with water and the unused sodium hydroxid titrated with double normal sulphuric acid, using litmus as indicator.

When analyzing solutions containing less than 30 per cent of formaldehyde the mixture must be allowed to stand 10 minutes after adding the hydrogen peroxid to complete the reaction.

The percentage of formaldehyde is found by multiplying the number of cubic centimeters of soda solution used by 2 when 3 grams of substance are taken.

Acetaldehyde reacts much more slowly with the reagent than formaldehyde, and it is doubtful whether the reaction is quantitative. Paraldehyde reacts still more slowly with hydrogen peroxid, even in the presence of a trace of ferrous salt. Benzaldehyde is acted upon more quickly, particularly in presence of ferrous sulphate, and a considerable evolution of gas takes place, but a long time is needed to complete the reaction.

(2) Method in use at the Connecticut Agricultural Experiment Station. (See Twenty-third Annual Report, 1899, p. 143.)

NICOTIN.

(1) *Kissling's method.* See Wiley, Principles and Practice of Agricultural Analysis, Vol. III, p. 606.

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[1] "So fast is this double combination that when sodic chlorid is present, no permanent precipitate of silver chlorid occurs until the quantity of silver necessary to form the compound is slightly overstepped." Sutton's Volumetric Analysis, 1890, p. 173, which see for further details; also Fresenius Quantitative Analysis, 1886, p. 450.

[2] Ber. **31**, 2979.

(2) *Winton's modification.* See Bulletin 56, Division of Chemistry, United States Department of Agriculture, p. 125.

(3) *Loyd's method.* See Bulletin 56, Division of Chemistry, United States Department of Agriculture, p. 114.

The methods for fungicides and insecticides presented by Mr. Voorhees in his report were adopted provisionally for trial and comparison in actual analytical work.

The report of the committee on abstracts was now called for, and, in the absence of Mr. Allen, was read by Mr. Beal, as follows:

REPORT OF THE COMMITTEE ON ABSTRACTING.

Mr. President and Members of the Association:

The work of the committee on abstracting literature relative to methods and apparatus for agricultural analysis has progressed during the past year with a fair degree of regularity. As soon as possible after the designation of the committee, the journals accessible to its members were ascertained and the following allotment was made by the chairman:

E. W. Allen....Jour. Landw.; Milch Ztg.; Ztschr. Untersuch. Nahr. u. Genussmtl.; Ztschr. Physiol. Chem.; Chem. Centbl.; abstracts in Analyst and Chem. Ztg. Report.
J. T. Anderson..Chem. Ztg.; Jour. Chem. Soc. [London].
W. H. Beal.....Compt. Rend.; Bul. Soc. Chim. Paris; Oesterr. Chem. Ztg.; Ztschr. Angew. Chem.; Ber. Deut. Chem. Gesell.; abstracts in Jour. Chem. Soc. [London].
R. E. Blouin....Bul. Assoc. Chim. Sucr. et Distill.; Jour. Amer. Chem. Soc.
W. F. Hand....Chem. News; Jour. Soc. Chem. Ind.
G. W. Shaw....Amer. Chem. Jour.; Analyst.
Harry Snyder...Bul. Assoc. Belge Chim.; Rev. Chim. Analyt. et Appl.
J. P. Street.....Landw. Vers. Stat.; Jour. Franklin Inst.
C. B. Williams..Ztschr. Analyt. Chem.; British Food Jour.

It is inevitable that at times the work assigned to individual members of the committee will accumulate, since most of the members are actively engaged in laboratory work and are likely to have unusual pressure of work upon them at certain periods in the year. If the work accumulates to any considerable extent, it is likely to become a burden upon the abstractor and to result in the oversight of part of the articles. To avoid this and to keep the work up to date, the plan has been followed, as in the past, of having such numbers of journals as have become two months in arrears abstracted in the Office of Experiment Stations. The object of this, as will be evident, is merely to prevent the work becoming burdensome and to avoid gaps.

During the past year there has been quite the usual amount of investigation on chemical methods, some of which it may not be without interest to mention very briefly, with the reference to the place of publication of the abstract in Experiment Station Record (E. S. R.)

The discovery of the occurrence of perchlorate in Chile saltpeter and its injurious effect upon crops has brought out a considerable number of articles on the frequency and extent of its occurrence, its injurious effects, and the methods for estimating it (E. S. R., XI, p. 110).

The constitution of the ammonium-magnesium phosphate of analysis has been reported upon in a valuable contribution by F. A. Gooch and Martha Austin (E. S. R., XI, p. 107).

F. P. Veitch has investigated the direct determination of available phosphoric acid and pointed out the cause of the discrepancy between the results by the Ross direct method and the official method (E. S. R., XII, p. 306).

E. Hotter has studied the solubility of Thomas slag in citric-acid solutions and recommends the use of a solution not stronger than 4 per cent (E. S. R., XI, p. 109).

Aschman has described a method for determining the total phosphoric acid in Thomas slag, using nitro-sulphuric acid as a solvent (E. S. R., XI, p. 507).

W. Hoffmeister has reported the results of studies with various slags, which indicate that his humic-acid method furnishes a reliable means of determining the relative value of different phosphates (E. S. R., XI, p. 1104).

Maxwell has reported his investigations on the estimation of available lime, potash, and phosphoric acid in soils by means of aspartic acid (E. S. R., XI, p. 507).

J. Plot has described the determination of phosphoric acid available as plant food in soils and fertilizers by means of a solvent, which it is claimed closely resembles beet juice in respect to salts. He gives a formula for its preparation (E. S. R., XII, p. 211).

Hess and Campbell have reported a new method for the direct determination of alumina in the presence of iron, manganese, calcium, and magnesium (E. S. R., XI, p. 611).

H. Lasne has given detailed directions for microscopical and chemical examination of bone superphosphate for purposes of detecting adulteration (E. S. R., XI, p. 104).

C. M. Van Deventer has proposed a new method of determining nitrates based upon the formation of brown solutions by the reaction between nitrates and ferrous sulphate (E. S. R., XI, p. 811).

E. W. Bell described a method for estimation of potash in fertilizers, soils, and vegetable substances, which is reported as having given very satisfactory results (E. S. R., XI, p. 109).

The determination of humus in soils has been described by C. Aschman and H. Faber (E. S. R., XI, p. 110).

A new method for the determination of clay in soils, by F. Poquillon, is described as fully as accurate and much more rapid than former methods (E. S. R., XI, p. 903).

Thorn Smith has proposed a method for the estimation of arsenic in paris green which reduces the time required for analysis to less than an hour (E. S. R., XI, p. 614).

T. B. Stillman has studied the variation in the composition of paris green and proposed a scheme for its analysis (E. S. R., XI, p. 22).

O. Kellner, F. Herring, and O. Zahn have tested König's method for the determination of pentosan-free crude fiber (treatment in an autoclave with sulphuric acid and glycerin). The crude fiber by this method contained from 2 to 3 per cent of pentosans, whereas that by the Weende method contained from 16 to nearly 20 per cent (E. S. R., XI, p. 704).

A critical study of the inverting power of tartaric, citric, and oxalic acids upon sucrose has been reported by H. Gillott (E. S. R., XI, p. 20).

The determination of cane sugar in condensed milk has been investigated at considerable length by L. Grünhut and S. H. Riiber (E. S. R., XII, p. 211).

P. Chapelle has described a new method for the gravimetric determination of reducing sugars, based upon the use of the centrifuge (E. S. R., XII, p. 106).

M. D. Crispo has described a new process for the determination of starch, based upon the polarization of a solution of the starch in potash, which, however, has not been tested on cereals (E. S. R., XI, p. 417).

Directions for the qualitative and quantitative examination of flour, with especial reference to adulteration with corn meal, have been given by H. Kraemer (E. S. R., XI, p. 211).

J. K. Haywood has worked out a modification of the Brücke method for determining glycogen, which is sufficiently accurate for distinguishing horse meats from other meats (E. S. R., XII, p. 107).

Upon the subject of milk and food preservatives, especially boric acid and formaldehyde, there have been a large number of articles treating of the extent of their use, the means of detection, and their effect upon the human system (E. S. R., XI, pp. 184, 212, 213, 312, 418, 419, 482, 575, 587, 618, 705, 769, 904, 960, 962, 984; XII, pp. 213, 214, 280).

There has been a large amount of work on the detection of adulteration in butter, the range in the constants with the change of period of lactation and with the change of feed (E. S. R., XI, pp. 111, 975; XII, pp. 18, 181, 308).

Among the new apparatus, perhaps the most important is that for ash determination described by Shuttleworth (E. S. R., XI, p. 304) and modified by Tucker (E. S. R., XI, p. 506). This apparatus, with the use of acetate of lime to prevent the formation of silicates insoluble in hydrochloric acid, places in the hands of the analysts a means for increasing the accuracy of ash determination and ash analysis. The investigations which these two gentlemen have reported show how relatively large may be the losses and the errors by the more common and cruder methods of incinerating materials.

An electrical method for the determination of nitrogen in organic substances has been described by Budde and Schou (E. S. R., XI, p. 306), in which the digestion in the Kjeldahl flask is accelerated by means of an electric current. The method does away with the addition of any foreign substance to promote the reduction. The results reported by these authors show a rather closer agreement with the theoretical than the ordinary Kjeldahl method gave.

A. W. Blyth has devised an apparatus in which nitrites and nitrates, either singly or together, can be determined as nitric oxid by means of ferric chlorid (E. S. R., XI, p. 21).

J. L. Hills, B. O. White, and C. H. Jones have reported the results of investigations on the experimental error involved in sampling crops for analysis (E. S. R., XI, p. 307).

C. A. Browne, jr. (E. S. R., XI, pp. 308, 615, 616) has given a valuable contribution to the chemistry of butter fats, dealing with the physical and chemical constants, the chemical composition, and the chemistry of rancidity in butter fat.

T. B. Osborne has continued his valuable investigations on the proteids of agricultural plants and products (E. S. R., XI, p. 309; XII, No. 6).

H. Droop Richmond has given us a very valuable contribution to the chemistry of dairy products in his Dairy Chemistry: A Practical Handbook for Dairy Chemists and Others Having Control of Dairies (E. S. R., XI, p. 618).

These are only a few of the papers relating to methods of agricultural analysis which have been noted by the abstract committee and abstracts published currently in the Experiment Station Record.

E. W. Allen,
J. T. Anderson,
W. H. Beal,
R. E. Blouin,
W. F. Hand,
G. W. Shaw,
J. P. Street,
H. Snyder,
C. B. Williams.
Committee.

The report of the committee on fertilizer legislation was asked for at this stage of the proceedings, but no report was made, the committee having taken no new action in the matter.

Mr. Huston suggested that the report already submitted was a good starting point.

Mr. Wiley agreed to have reprints made of the report.

The President. The Association of Commissioners of Agriculture of the South presented the views of this committee and urged the commissioners to adopt them. The chemists should seek the aid of these gentlemen.

The report of the committee on recommendations was then presented.

REPORT OF COMMITTEE ON RECOMMENDATIONS.

SUGAR.

The committee recommends that the referee for the ensuing year study the method of Clerget and the German method.

Adopted.

REPORT OF COMMITTEE ON VOLUMETRIC STANDARDS.

The report of the committee on volumetric standards was presented by Mr. Ewell, at the request of the president. It consisted in a statement of the progress made in the movement to establish a national standardizing bureau, and proposed the following resolutions, which were adopted:

Resolved, That the Association of Official Agricultural Chemists most heartily indorses the movement in progress for the establishment in this country of a national standardizing bureau, and hereby declares that the absence of facilities such as would be provided by the proposed bureau has seriously hampered the work of this association, owing to the difficulty of obtaining in this country, with official certificates of accuracy, the flasks, burettes, pipettes, weights, thermometers, polariscopes, and other apparatus needed in the work of official chemists. The use of apparatus which bears the official stamp of the Government would eliminate one element of dispute in commercial analyses, thus preventing the expense of litigation, and would in general increase the value of the work of this association by facilitating the attainment of uniform results.

Be it further resolved, That copies of these resolutions be forwarded by the members of this association to the Senators and Representatives representing their respective States in Congress.

B. W. Kilgore, *President.*
H. W. Wiley, *Secretary.*

Mr. Williams here presented a method of marking flasks by the use of paraffin and beeswax.

Mr. Frear then presented the report of the committee on food standards, as follows:

REPORT OF COMMITTEE ON FOOD STANDARDS.

On behalf of your committee on pure-food standards I would report that a meeting was held in Washington in March last, at which were present most of the members of the general committee, as well as a large number of the referees, who are collaborating with them and whose counsel in the final deliberations of the committee was most helpful.

The general plan of operation adopted by your committee makes it necessary to delay the announcement of the details of the work until the latter shall have reached some measure of completeness. This plan involves the preparation by the several referees and the consideration by your committee of correct definitions and classifications of food materials and the consideration of proposed standards in the light of American experience, as shown by the work of your committee and the referees and by compilations of existing data from materials of known character.

This preliminary work done, it is planned that the definitions and various suggested standards for the several classes of products be submitted to competent representatives of the respective trade interests concerned for criticism and suggestion before any publication of the conclusions is made by the committee, to the end that no overhasty action to the unfair detriment of any interest be taken and that the completed work may find general acceptance on the ground of its scope, accuracy, fairness, and unity.

To give some general notion of the work done, I may, however, state that definitions and standards have been tentatively adopted for the following classes of products:

Definitions: Meats (fresh, preserved, pickled, salted, and smoked); sausages; lard; milk; skim milk; cream; butter; buttermilk; cheese; whey; koumys; condensed milk; pasteurized milk; ice cream; fruit butter; jam, marmalade and fruit jelly; sugar; maple sugar; molasses; sirup; glucose sugar; glucose sirup.
Standards: Lard; milk; butter fat; butter; cream; cheese; skim milk; condensed milk; fruit butter; jam, marmalade preserves, and fruit jelly; sugar; molasses; sirup; glucose sugar; glucose sirup; flavoring extracts of cinnamon, peppermint, lemon, and orange.

Sets of definitions and standards have been offered for consideration by the referees, but not yet formally acted upon by the committee, for the followings substances:

Definitions: Salt; olive oil; spices; tea; coffee; cocoa; chocolate; fruit juices; fermented cider; perry; wines; vinegars; malted liquors; spirituous liquors.
Standards: Salt; olive oil; spices; tea; coffee; cocoa; chocolate; fermented cider; dry white wine; dry red wine; fortified sweet wine; cider vinegar; wine vinegar; malt vinegar; malt beer; malt extracts; whisky; brandy; rum; gin.

In connection with this work the following collections of data, in many instances involving original research, have been made for the specific work of this committee, and those not yet presented for publication are now nearly ready for the press:

(1) *Lard.*—Results of analyses of lard used for cheese filler. By J. B. Weems.

(2) *Milk products.*—Milk: Compilation of averages of normal milk varying between 3 and 5 per cent of butter fat, representing 5,552 samples. By L. L. Van Slyke.

(3) *Cocoanut oils.*—Results of analyses of cocoanut oils used as butter surrogate. By J. B. Weems.

(4) *Grains and their products.*—Computation of maximum, minimum, and averages representing the composition of barley meal and pearl barley; buckwheat products—flour, self-rising buckwheat, prepared buckwheat, buckwheat farina; corn products—meal, granular, unbolted, flour, velvet meal, cerealine, hominy, New Mexico corn, pop corn (raw and popped); prepared infant and invalid foods (16 kinds); oat breakfast foods (15 kinds); macaroni and vermicelli; wheat breakfast foods (26 kinds); wheat flour (20 kinds); bread and other baked flour products (51 kinds); starches (2 kinds). By W. O. Atwater.

(5) *Cattle foods.*—Compilation of 636 complete or partial analyses of 16 kinds of concentrated cattle foods. By J. B. Lindsey.

(6) *Fruits and vegetables.*—A compilation of analyses of market fruit butters, jellies, preserves, and marmalades, representing 38 samples. By H. A. Weber.

(7) *Condiments.*—A compilation showing (1) standards recommended for various species by Vogl and Hanausek, published in Codex Alimentarius Austriacus; (2) results of 206 American analyses of species and their adulterants, the majority of them by the compiler and his associates, together with description of microscopic and chemical methods of investigations. By A. L. Winton.

(8) *Fruit extracts.*—Results of analyses of fruit extracts for table use. By H. A. Weber.

(9) *Salt.*—Compilation of 87 American analyses of table, dairy, and packing salts, largely made by the compiler for use of the committee. By F. W. Woll.

(10) *Olive oil.*—Compilation of the properties of olive oil, European and American, and of its salad-oil substitutes and adulterates, including the index of refraction, specific gravity, thermal degree, viscosity of soap solution, iodin number, saponification value, melting point of fatty acids, behavior under special tests: Brullé, Bechi, Schneider, Renard, Baudouin, and Hauchecorne, including detailed results. By G. E. Colby.

(11) *Cotton-seed oil.*—Results of analyses of cotton-seed oil. By J. B. Weems.

(12) *Beverages and vinegar.*—Compilation of American and European results of analyses of apple juice, fermented cider, cider and spirit vinegar. By William Frear.

Your committee desires to express its gratitude for the able and painstaking collaboration of the several referees and their associates in this work, to whom the progress it has thus far made is chiefly due.

The committee also desires to express to Hon. James Wilson, Secretary of Agriculture, its appreciation of his cordial indorsement of their work and the aid he has given in facilitating correspondence by appointing the members of the committee and the referees in charge of main classes of subjects special agents of the United States Department of Agriculture.

In closing this report of its work your committee requests that it be continued for further effort toward the end proposed.

Respectfully submitted in behalf of the committee.

William Frear,
Chairman.

The report was adopted and the committee continued for the ensuing year.

The following resolution was presented by Mr. Frear for the committee on pure-food standards:

Resolved, That the Association of Official Agricultural Chemists of the United States hereby reaffirms its conviction of the urgent necessity for national legislation on broad lines for the prevention of adulteration of foods and drugs that are subjects of interstate commerce and commerce with foreign lands, for the better protecting of the health of our citizens and the legitimate interests of producers and dealers, and for the national reputation of business honor, and most earnestly and respectfully urges upon the Congress of the United States the present enactment of the measure now on the Calendar of the House of Representatives with a favorable recommendation, after long and careful consideration of the Committee on Interstate and Foreign Commerce, which is essentially the bill introduced in the present Congress in the House by Hon. Marriott Brosius, of Pennsylvania, known as H. R. 9677, and in the Senate by Hon. H. A. Hansbrough, numbered 2222 and now under consideration by the appropriate committee of that body.

Resolved further, That the secretary of the association be directed to convey to the proper officers of the Congress of the United States copies of these resolutions.

Adopted.

The report of the committee on resolutions was then called for, which was presented, as follows:

REPORT OF COMMITTEE ON RESOLUTIONS.

Resolved, That the Association of Official Agricultural Chemists return cordial thanks to the Secretary of Agriculture, the authorities of Columbian University, and the governing board of the Cosmos Club for the courtesies they have shown to the association.

Adopted.

The PRESIDENT. I am sorry we have not the blackboard before us, so we can see the roll of this meeting. It numbers upward of 85. This is the largest attendance this association has ever had. Dr. McMurtrie, president of the American Chemical Society, in speaking to me to-day, said, "If we can have 100 members present at the Chicago meeting of the American Chemical Society, I shall consider that we are very fortunate." Inasmuch as we are but a small part of the broad field of chemistry, I feel that the good showing at this meeting is a matter for congratulation. Another matter has come before us at this time. We have had our friends from across the border come to the association with their troubles. They recognize the value of our methods. We are glad to hear from the report of our secretary that our methods are appreciated and recognized, not only abroad, but also at home their great value is seen. These things make me appreciate all the more the great honor you have done me in selecting me to preside over the seventeenth annual convention, and I thank you most heartily and sincerely for this compliment.

If there is no further business, I shall declare the seventeenth annual meeting of the Association of Official Agricultural Chemists adjourned sine die.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1901.

PRESIDENT.

Mr. L. L. VAN SLYKE, Geneva, N. Y.

VICE-PRESIDENT.

Mr. H. J. WHEELER, Kingston, R. I.

SECRETARY.

Mr. H. W. WILEY, Washington, D. C.

ADDITIONAL MEMBERS OF EXECUTIVE COMMITTEE.

Mr. W. R. PERKINS, Agricultural College, Miss.
Mr. F. W. TRAPHAGEN, Bozeman, Mont.

REFEREES.

Phosphoric acid.—Mr. H. K. Miller, Lake City, Fla.
Nitrogen.—Mr. W. R. Perkins, Agricultural College, Miss.
Potash.—Mr. C. L. Hare, Auburn, Ala.
Soils.—Mr. M. E. Jaffa, Berkeley, Cal.
Dairy products.—Mr. J. A. LeClerc, Geneva, N. Y.
Foods and feeding stuffs.—Mr. W. H Krug, Washington, D. C.
Liquor and food adulteration.—Mr. W. D. Bigelow, Washington, D. C.
Sugar.—Mr. E. E. Ewell, Washington, D. C.
Tannin.—Mr. W. K. Alsop, 26 Ferry street, New York.
Insecticides.—Mr. Louis A. Voorhees, New Brunswick, N. J.
Ash.—Mr. G. S. Fraps, Raleigh, N. C.

ASSOCIATE REFEREES.

Phosphoric acid.—Mr. C. H. Jones, Burlington, Vt.
Nitrogen.—Mr. Fred W. Morse, Durham, N. H.
Potash.—Mr. H. B. McDonnell, College Park, Md.
Soils.—Mr. F. P. Veitch, Washington, D. C.
Dairy products.—Mr. George Cavanaugh, Ithaca, N. Y.
Foods and feeding stuffs.—Mr. C. A. Browne, jr., State College, Pa.
Liquor and food adulteration:

(1) Meat and fish.—W. D. Bigelow, Washington, D. C.
(2) Fats and oils, not including dairy products or their substitutes.—L. M. Tolman, Washington, D. C.
(3) Cereal products.—A. McGill, Ottawa, Canada.
(4) Infants' and invalids' foods.
(5) Saccharine products, including confectionery.—A. E. Leach, Boston, Mass.
(6) Vegetables—canned, dried, or otherwise preserved.—L. S. Munson, Washington, D. C.

(7) Tea, coffee, and cocoa.
(8) Spices and condiments.—A. L. Winton, New Haven, Conn.
(9) Vinegar.—William Frear, State College, Pa.
(10) Flavoring extracts.—A. S. Mitchell, Milwaukee, Wis.
(11) Fruit products.—L. M. Tolman and L. S. Munson, Washington, D. C.
(12) Fermented and distilled liquors.
(13) Baking powders and baking powder chemicals.—A. L. Winton, New Haven, Conn.
(14) Preservatives.—W. M. Allen, Raleigh, N. C.
(15) Dyes.—L. M. Tolman, Washington, D. C.

Sugar.—Mr. G. L. Spencer, Washington, D. C.
Tannin.—Mr. W. H. Teas, Ridgway, Pa.
Insecticides.—Mr. J. K. Haywood, Washington, D. C.
Ash.—Mr. E. W. Magruder, Richmond, Va.

COMMITTEE ON ABSTRACTING.

Mr. E. W. Allen, Washington, D. C., Chairman.
Mr. W. H. Beal, Washington, D. C.
Mr. J. T. Anderson, Auburn, Ala.
Mr. J. P. Street, New Brunswick, N. J.
Mr. W. F. Hand, Agricultural College, Miss.
Mr. Harry Snyder, St. Anthony, Minn.
Mr. C. B. Williams, Raleigh, N. C.

COMMITTEE ON FOOD STANDARDS.

Mr. Wm. Frear, State College, Pa., Chairman.
Mr. H. W. Wiley, Washington, D. C.
Mr. H. A. Weber, Columbus, Ohio.
Mr. M. A. Scovell, Lexington, Ky.
Mr. E. H. Jenkins, New Haven, Conn.

COMMITTEE ON FERTILIZER LEGISLATION.

Mr. H. W. Wiley, Washington, D. C., Chairman.
Mr. B. W. Kilgore, Raleigh, N. C.
Mr. H. B. McDonnell, College Park, Md.
Mr. H. A. Huston, Lafayette, Ind.
Mr. B. B. Ross, Auburn, Ala.

COMMITTEE ON VOLUMETRIC STANDARDS.

Mr. B. W. Kilgore, Raleigh, N. C., Chairman.
Mr. C. L. Penny, Newark, Del.
Mr. E. E. Ewell, Washington, D. C.
Mr. G. C. Caldwell, Ithaca, N. Y.
Mr. H. W. Wiley, Washington, D. C.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members, and to have all privileges of membership, save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a referee and associate referee for each of the subjects to be considered by the association.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same, to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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